Technological Controls in Leather Manufacture

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PREFACE

Modern technology aims at improving both quality and quantity of the product. It is the mark of quality that reflects reputation, ready consumer acceptance and competitiveness in internal and external markets. Quality control starts with the raw material inputs through each unit process in the production line to the final product. Process and product controls are thus basic ingredients of modern technological production.

The theme of quality control and standardisation assumes greater significance in the leather industry in as much as the natural raw material—hide or skin is non-uniform in size, shape, thickness, weight, fibre texture etc. They vary with age, sex, breed, feed source etc. of the same animal. The process of leather making is essentially a sustained series of eqilibrium reaction, each affected by the previous and subsequent reactions. Each reacting agent and operation affects both the structural, physical and chemical properties. Thus, a judicious adjustment of methods and materials are needed to tailor the molecule to meet a wide variety of end uses of leather, this complexity is the despair and delight of the tanner. All the greater is the need for stricter standardisation of the purity of input materials, maintaining physical parameters such as float, temperature, pH, drum speed etc. Only then pack-to-pack uniformity in the production can be ensured.

This booklet outlines the tests that could be carried out during the various unit processes towards standardisation of the production. The procedures for conducting such tests are dealt with in such a way that they can be understood and put into practice with ease. The important characteristics of some specific types of leathers are indicated in the second Chapter. Actual testing and analysis to determine various characteristics of leathers including the sampling position are given in Chapter three. It must, however, be understood that these characteristics alone do not fully reflect the properties of leathers; other intrinsic properties such as feel, break, fullness, softness, smoothness of grain etc. in required degrees are also important to have a complete assessment of different

types of leathers. References to literatures on this subject, recommended quality requirements for the main types of leathers and addresses of suppliers of physical testing instruments are also provided.

Greater efforts by those engaged in the fields of quality control and standardisation and the industry are needed towards improving the production on modern scientific and technological lines. This booklet will be of great help to the industry, both in the rural as well as organised sector. This is a timely publication in the context of combined efforts of the Govt. of India and the industry towards raising the unit-value of Indian leathers. It is hoped that the industry makes full use of the information contained in this NICLAI publication with an eye on international reputation for Indian leather quality.

Y. Nayudamma

FOREWORD

An important requirement for marketing is the quality of products being marketed. This very well applies to leathers as well and more so, as it is an internationally marketed commodity. In actual leather manufacture, quality control of raw materials, processes and products is essential, particularly when one is dealing with a hetrogeneous raw material such as hide or skin and competing in international markets.
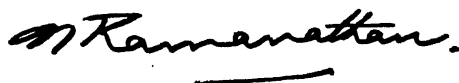
Quality control and standardisation become the watchwords. Standardisation is taken to mean agreement upon and ʾdefinition of performance, quality, composition, dimensions, method of manufacture and/or testing of a product.

In recognition of the importance of quality control and standardisation, this monograph "Technological Controls in Leather Manufacture" is being published for the benefit of the leather industry.

This monograph contains information on a wide range of very useful and simple tests that can be applied during the processing of leathers, starting from raw materials, process controls and testing/analysis of leathers for product control. Important physical and chemical characteristics of different types of leathers are indicated. Mention is also made of the applications of tests relevant to different types of leathers. Extracts from the UNIDO publication on "Acceptable Quality Levels in Leathers" highlighting the recommended requirements for the main types of leathers have also been reproduced here to focus the international needs. References to official methods of testing provide additional useful information.

Bibliographical references and the list of suppliers of physical testing equipment given in the monograph may prove helpful to set up a control laboratory in the tanneries.

It is sincerely hoped that industry will be greatly benefited by making use of this handy publication.

M. Ramanathan.

(ACTING DIRECTOR)

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INTRODUCTION

Animal hides and skins form the basic raw materials for the production of leathers. "Hide" is the "skin" of a big animal like cow and buffalo, and "skin" is the "hide" of a small animal like goat and sheep. The readily putrescible raw hide or skin is processed and tanned into "leather" which is more resistant to heat, hydrolysis, and micro-organisms. During this process, the skin protein molecule is tailored to meet the variety of end-uses of leather.

Though their chemical composition is similar, the raw hides and skins vary in thickness and structure. There is much variation even within the skin of the same animal depending on the anatomical locations of the skin. The fibre weave of the skin from the neck and butt areas is compact and tight while that of the belly region is loose. The leatherware manufacturers have to be extremely cautious in selecting the proper varieties of leathers taking into account the locational variations within a single piece of skin. Different end-uses dictate different requirements in the leathers. Thus, leather panels for football should have not only a minimum but also equal amounts of elongations when pulled in more than one direction; a leather belting must have grip, less stretch and uniform thickness; gloving leather should have good elongation, stretch, and resilience; garment leather should have comfort factors, sufficient porosity, and drape; sole leather must be resistant to wear, tear, water, etc., and should be solid and compact.

Different end-uses and different requirements need different tests—visual, physical and chemical—on the leathers. As leather is a non-uniform product, performance test is found to be the best in practice. Physical and chemical tests and the relevant standard specifications will be more in the way of indicators and do not necessarily correctly correlate to the performance tests. The various tests, other than the performance tests, are only suggestive of the performance of the leathers and the components of a particular leather article; the resultant performance of the leather article as such is decided by a combination of all these various properties tested. The results of tests on leathers thus serve more as guides to understand beforehand the likely performance of the leather articles to be fabricated out of them and to help select suitable pieces of leather for such given type of fabrication. As the grading of the leathers is based on a non-uniform pattern, the norms being different among different trade transactions—both national and international—the quality of leathers that are prescribed for testing will greatly vary and the test results

have to be read with a certain amount of restraint and should, as a rule, be confined to the particular pack and the scale of sampling as accepted between the buyer and the seller.

Attempts are made to correlate physical and chemical characteristics of leathers. In the case of vegetable tanned buffalo hides, while the oils and fats, fixed tannins, leather substance and the degree of tannage are found to be directly proportional to the tensile strength and tearing strength, they are indirectly proportional to water absorption. This trend is reversed in the case of water solubles against these physical characteristics.

The first chapter of this monograph deals with some simple tests that would be helpful to the tanners during the production of leathers and gives useful hints on some non-destructive testing techniques based on visual and simple tests that could be applied with advantage on leather articles as well as on leathers themselves. The second chapter outlines important characteristics of certain specific leathers. A detailed account of the test procedures and their areas of application are provided in the third chapter. The recommended quality requirements for the main types of leathers and an indicative list of suppliers of physical testing instruments are given in the appendix.

USEFUL TESTS TOWARDS PROCESS CONTROL AND STANDARDISATION IN LEATHER PRODUCTION

The raw hide or skin goes through a series of equilibrated reactions and processes, both chemical and mechanical, before it is tanned into leather, finished and made ready for end-use. In this chapter, a few simple and quick tests of leathers and processing materials which serve as useful hints to the tanners for process control are described. This is followed up with a note on some of the non-destructive tests on leathers and leather articles.

RAW HIDES AND SKINS

Hides that are not properly cured can be identified by bad odour and hair-slip. Even the well-cured hides, during transit in summer months, may undergo heat damage due to insufficient protection and uneven drying. Such hides may apparently look and smell proper but will not have any puncture strength when tested with a pencil, nail or screwdriver.

NATURE OF WATER

Water supplies in the tannery should be checked for hardness, salt content and iron contamination; such testing will help locate the origin of most of the troubles confronting the tanners. Hard and saline waters will result in loss of vegetable tannins, patchiness in dyeing, breaking of fatliquor emulsions, etc. Hardness also reduces the extent of swelling and plumping of hides and skins during liming leading to lower yields of final leathers. Iron is highly detrimental to vegetable tanning.

PRETANNING PROCESSES

While using *sodium sulphide*, it should be ensured that the sulphide is completely dissolved and cooled before application; undissolved solids can cause grain pits and weak grain in the areas of contact and can lead to poor dye uptake.

When ammonium sulphate, containing impuiities such as lead, nickel and cobalt, is used for *deliming*, brown stains with or without black speck marks may be formed on the leather; to detect such contamination one can treat a solution of the suspected ammonium sulphate (one teaspoonful of ammonium sulphate in one half cup of water) with one half-teaspoonful of good quality sodium sulphide; the formation of black colour indicates contamination.

The use of phenolphthalein to check the *extent of deliming* and *preparing* the pelt *for bating* must be scrupulously followed and not ignored lightly.

It is a common practice in the tanneries these days to conduct the *pickling* in low floats. Utmost care is required to avoid acid burns; the excessive heat developed during the addition of the acid, particularly sulphuric acid, causes such burns. Considering the magnitude of this type of damage, which is easily avoidable, it may even be preferable to make pickle baths one day earlier.

Grain damage can be distinctly seen on acetone-dehydrated pelt and leather pieces. The pelt, vegetable tanned leather or wet blue leather is smooth when moist. After the acetone dehydration, the exposed fibres, so exposed due to the removal of grain enamel, give a rough feel to the touch.

Acetone dehydration also helps in detecting pickle acid burns and other *heat burns* due to live steam over the leather surface; the leathers after the acetone dehydration, when dried in an air oven, develop cracks in the grain resembling a broken glass pattern.

CHROME TANNING

Checking for thorough *penetration* of chrome during wet blue chrome tanning is of paramount importance as otherwise, any untanned streak in the middle will lead to difficulties during the subsequent unit operations and ultimately gets reflected in the final leathers. Only after the chrome had penetrated through the entire cross-section of the leather, the basification should be started.

Determination of the *shrinkage temperature* (hydrothermal stability) of the leathers during tanning is suggested to ascertain the extent of tanning; more useful and meaningful observations are made by finding the shrinkage temperatures in the case of chrome leathers as compared to the boil-test.

Many tanners still prefer to make their own chrome liquors using molasses or sugar for reducing the dichromate. Standard procedures are to be followed for batch-to-batch production. Besides checking for complete reduction of the dichromate, the absence of burned sugar should also be ensured in the liquors; burned

sugar, if present in liquors gets deposited on the surface of wet blue leathers making them look brown instead of the characteristic wet blue colour. The presence of *burned sugar* may be checked by washing a small quantity of chrome powder or liquor placed on a white filter paper in a funnel with dilute sulphuric acid until the chrome is removed; brownish colour will be imparted to the paper in the presence of burned sugar.

Iron contamination or rust will cause *pink stains* with chrome and formate. To test for iron contamination, stained as well as the non-stained portions of the leather surface are treated with a small quantity of dilute hydrochloric acid followed by a dilute solution of potassium ferrocyanide. If the pink surface turns blue, contamination with iron is confirmed. In the case of vegetable tanned leathers, iron contamination leads to greenish blue to dark stains and it can be confirmed in the same way with potassium ferrocyanide.

Black stains on leathers are mostly attributed to the contamination with lead which might have occurred during the transportation of raw hides and skins in dirty lorries and trucks. The lead reacts with sulphide during the unhairing process forming lead suphide; this lead sulphide is not soluble in acid and persists in the finished leather. To detect lead contamination, a dilute solution of sodium chlorite may be placed on the stain. The stain will turn orange, due to the formation of lead chromate, if lead contamination has occurred. This test is applicable only for chrome tanned leathers.

Dark green stains on the leathers are the result of chrome precipitation. These can be removed by gently acidifying the leathers with organic acid or dilute sulphuric acid and leaving overnight followed by basification with sodium bicarbonate cautiously. Such stains may also result from the presence of grease in greasy hides still left after incomplete degreasing after pickling; these hides are more sluggish during neutralisation and chrome gets deposited on the surface. These dark green stains can be cleared as above and may also be avoided by adding sodium bicarbonate more slowly.

DYEING, FATLIQUORING AND FINISHING

To check the *hues and tones of the dye colours,* filter paper strips are dipped in dilute solutions of the dyes and the paper strips observed carefully.

The '*Blow-out*' test helps to find out whether a dye contains one or more components. A small amount of the dye taken at the tip of a knife blade or spatula is blown on to a large piece of damp filter paper when the separated particles dissolve in the water present in the paper and produce one or more colours depending on the number of component(s) present in the dye.

Dyes should be checked for *sensitivity to alkali, acid and iron*. For this a filter paper is taken and four spots of a dilute solution of the dye are placed on it. The first spot is treated with dilute sodium bicarbonate, the second with dilute acetic acid, the third with dilute ferric chloride, leaving the fourth as the control. Any change in the colour of the first or second spot as against the control indicates possible difficulties with the pH of the tannery water. If the third spot changed colour, the iron, if any, in the tannery water may be expected to give problems. If the dye changed colour with acid or base, the exact pH at which the change occurs is determined with the help of a pH meter; dyes having such colour changes at pH values between 3.5 and 4.5 may be avoided as they may not give uniformity of colour.

To test the *charge of the dyes*, paper chromatography can be used. Dilute hydrochloric acid and dilute sodium hydroxide (each 20 ml) are taken in two separate bottles. Paper strips that have been spotted with the dye solution are then suspended in each of the bottles such that the dye spot is above the liquid level; the movement of the dye spot is then observed. If the dye spot moves with acid, the dye is basic. If it moves with base, it is acidic.

Similar test can be conducted for *oil-soluble dyes* using chloroform in the place of acid and alkali. If the dye migrates up with chloroform it could be an oil-soluble dye. Since oil-soluble dyes stay with fatliquor rather than completely fixing with leather fibres and tend to diffuse into the finish of finished leathers, the resultant leathers, both suedes and full-grain leathers, will have poor rub fastness properties.

The *ionic charge of the leather surface* can be checked with a two-dye solution. The solution is a mixture of an acid dye (Orange-G, 50 mg) and a basic dye (Crystal violet, 2.5 mg) in solution with 95 per cent ethanol (90 ml) and water (10 ml). This solution is placed over the surface of leather in a particular place successively. If there is a yellow spot with a purple ring, the charge of the leather surface is cationic. If there is a purple spot with a yellow ring, the charge of the leather surface is anionic.

Greasy patches on leathers can be identified by gently rubbing them with a cloth dampened with chloroform and observing whether the patches are removed by such treatment. Leathers collect greasy stains sometimes when grease is splattered from machinery.

The *poor uniformity of oil take-up* can be checked by washing the surface of a cutting of crust leather with chloroform.

The *spue formation* on the leather surface results in considerable difficulties during finishing and the spues show up on the finished leathers as well. This

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problem is attributed to the poor quality of fatliquors and top dressings. The spue components can be determined in a simple way. The fatliquor (10 ml) is mixed thoroughly with ethyl ether (50 ml, fresh and free from hydrogen peroxide) and water (50 ml). After shaking well, the ether layer is allowed to come to the top. (Sodium chloride may be added if the emulsion does not break up.) While the water-soluble components of the sulphated or sulphonated oils stay in water, the spue components get dissolved in ether. The ether extract is taken off and the ether evaporated. The residual oil is then warmed in 50 ml of 95 per cent ethanol and then left over a period of several hours each day upto seven days at 20°C. If the crystals of fats form on the sides of the bottle or beaker, the sample of fatliquor can be suspected to cause spue problem.

Salt spue on chrome tanned leathers is usually caused by the formation of calcium sulphate through the reaction between the calcium from lime used for dehairing and the sulphate of ammonim sulphate used during the processing. The deposition of calcium sulphate is more significant if the leather undergoes only limited washings since the solubility of this salt is poor. To identifiy this, the spue is scraped into a small container to which are added a few drops of murexide reagent (a few milligrams of murexide in 5 ml water and 20 ml 95 per cent ethanol) followed by a few drops of dilute sodium hydroxide; calcium, if present, will cause pink colouration; in the absence of calcium, the murexide remains purple.

The characteristics of a *finishing composition* can be assessed by painting the finish on a piece of glass. The finish should dry, without remaining wet or tacky. The film can be removed from the glass plate by running a razor blade between the film and the plate; the film is then examined for stretchiness and flexibility. Similar tests can be done on resin binders as well.

Sometimes it is difficult to determine the nature of stains in leathers visually; microscopical examination of such samples will help identify the nature of stain. The fat, bacteria, mould, calcium, etc., are assessed by making use of frozen cross-sections of the leather as follows:

Staining technique for *fats* consists in passing the frozen sections through 50per cent and 70 per cent alcohol (2-3 minutes in each) successively, treating with a saturated solution of Sudan IV (Scharlach R) in 70 per cent alcohol for a period of 15-20 minutes followed by washing in 50 per cent alcohol (2-3 minutes) and distilled water and mounting in glycerin jelly; the fatty matter is stained red and is clearly visible under the microscope.

Staining for *bacteria* and *mould* is carried out by treating the frozen sections in 0.5 per cent methylene blue for 10-20 minutes, washing in distilled water, dehydrating in 50 per cent, 70 per cent and absolute alcohol (2-3 minutes in each), clear-

ing in xylol and mounting in neutral Canada balsam (chrome-tanned and vegetable-tanned sections are immersed in 1 per cent potassium permanganate solution for 15—30 minutes, transferred to 2 per cent sodium metabisulphite solution until the sections are colourless, washed in distilled water, bleached with 5 per cent oxalic acid and again washed with two changes of distilled water and then taken for staining with methylene blue solution); bacteria and mould stain bluish violet against light blue background and are observed through a microscope.

To stain for *calcium*, the frozen sections are treated with 5 per cent silver nitrate solution for about 10-30 minutes, 0.5 per cent hydroquinone solution for 2 minutes and 2 percent sodium thiosulphate solution for 5 minutes (a thorough rinsing with distilled water must be done between these three treatments), rinsed again in distilled water, dehydrated through 50 per cent, 70 per cent and absolute alcohol, cleared in xylol and mounted in neutral Canada balsam. Calcium deposits stain deep brownish black and are clearly observed through a microscope.

The Prussian Blue test for the staining of *iron* consists in treating the frozen sections with 2 per cent potassium ferrocyanide solution (freshly prepared and filtered) for 5 minutes, adding an equal volume of dilute hydrochloric acid to ferrocyanide solution, mixing well and leaving the sections in the mixture for a further 15-20 minutes, washing in distilled water, dehydrating by passing through increasing strengths of alcohol, clearing in xylol, and mounting in neutral Canada balsam or in synthetic resin; iron is coloured blue (the Prussian Blue fades slowly in Canada balsam but is more stable in synthetic resin) and can be seen clearly through a microscope.

The *resistance* of the leather for *mildew* is an important characteristic to be determined. This, however, depends on the end-use of the leather as well as on the type of fungicide (whether water soluble or not) which may have been used in the treatment of the leather. Untreated control leathers as well as leathers treated with agents to promote mould resistance are smeared, sprayed and swabbed with mixed spore suspension of species of mould found in tanned leathers and the resistance of such leathers to the growth of mould evaluated.

NON-DESTRUCTIVE TESTS ON FINISHED LEATHERS

The tests described above are applicable during the production. There are certain non-destructive tests which could be applied with advantage on finished leathers and leather articles.

The *crackiness of the grain,* layer and *finish* layer can be checked by both single folding and double folding, grain outwards.

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The *non-adhesion of finish layer* consequent on improper finishing, the *grain* itself *separating* off the rest of the leather due to bacterial attack underneath the grain layer etc., may be tested by folding the leather, grain inwards over sharp angles when the *pipiness of the grain* can be observed.

A *good finishing* will not develop cracks when a blunt key is forcibly drawn over the flesh side of the leather (*Key test*).

An idea on the degree of *fastness* of the finish *to rubbing* can be had by just rubbing the finish with a cloth held over the index finger; the cloth may either be dry or made wet with water or other solvents with which the fastness is to be tested.

The *resistance* of the leather *to water* may be judged by placing a drop of water over the surface of the leather and observing the time taken for it to sink into the leather.

A quick and rough estimate of apparent density, as the ratio of the weight of a piece of leather to its volume, will help in obtaining information on its *cutting value*; leathers having equal weights and thicknesses will yield larger or smaller areas for cutting into components of leatherwares, depending on their respective lower or higher density values.

Small pinholes and weak spots which are not normally visible to a naked eye are detected by spreading the leather on a glass plate which is fixed on the top of a box. A powerful source of light fixed in the box just below the glass plate reveals these defects.

Non-destructive testing techniques such as ultrasonics can be applied to footwear and other leather articles to detect flaws, if any. These tests are quite useful for testing industrial boots which contain toe caps, plates, etc., made of metals and other leatherwares containing such inner metallic components.

GENERAL

In general, the purities of all the chemicals—sodium sulphide, lime, deliming agents, vegetable tannin extracts, chrome extracts, etc.—and sometimes even the performance characteristics of say, syntans, dyes, fatliquors, finishing auxiliaries, etc., are to be tested periodically. As regards raw hides and skins, a tabulation of the assortments obtained from different types of raw materials is helpful in the standardisation of processes which will help in the proper planning of product-mix. Other

controls include those in respect of pH, float, speed of the drum, duration of each unit operation, extent of uptake, analysis of effluents during each unit operation, etc. The determination of other physical parameters such as temperature and weight of the pack during different stages of processing is also essential. Careful recording of such parameters in respect of each unit operation, testing hardness and other characteristics of daily water supplies in the tannery, tabulation of analytical results of the exhaust baths of say, pickling, chrome tanning (which will eventually help in the recirculation of baths towards curtailing the volume of effluents), barkometer readings during vegetable tanning of heavy leathers, etc., are the approved systems of quality control measures and standardisation procedures during the production of leathers.

CHARACTERISTICS OF SPECIFIC TYPES OF LEATHERS

Leathers may be broadly classified, with respect to their end-uses, into the following groups:

1. Heavy and industrial leathers,

2. Light leathers,

3. Sportsgoods leathers and

4. Parchments.

Thus, belting leathers, picking band leathers, hydraulic leathers, etc., are industrial leathers; sole and harness leathers, etc. are heavy leathers; garment leathers, upper leathers, linings, etc. are light leathers; leather for football, cricket ball, etc. are sportsgoods leathers; leather for puppets, musical instruments, etc. are parchments. The characteristics of most of these leathers are covered by national and international standard specifications. In the absence of prescribed specifications for certain leathers, those characteristics as are agreed upon between the buyer and seller are stipulated on such leathers. Such also is the practice when the buyer needs certain special properties to be incorporated in the case of those leathers that are already covered by the respective specifications.

In general, the requirements, viz., the results of tests that are most appropriate to a given type of leather, are prescribed by the national and international standard specifications, i.e., these tests are set as the requirements keeping in view the nature of use to which the particular leathers are put to during their actual use in the form of leather articles. In fact, most of these tests and the testing instruments needed for conducting such tests are common to both leathers and leather articles. Table I lists various types of leathers and important physical and chemical characteristics to be tested for some specific types of leathers.

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TABLE I

Important characteristics of different types of leathers

Type of leather	Characteristics to be looked into	
	Physical characteristics	Chemical characteristics
Upper leather	Tensile strength, elongation, tearing strength, stitch tear strength, air permeability, water vapour permeability, flexural endurance, scuff resistance, fastness to dry and wet rubbing, grain crackiness strength	Oils and fats, water solubles, hide substance, Cr_2O_3, fixed organic matter
Lining leather	Tensile strength, elongation, tearing strength, stitch tear strength, air permeability, water vapour permeability, water absorption, fastness to dry wet and rubbing (for coloured linings)	Oils and fats, water solubles, hide substance, Cr_2O_3, degree of tannage (for vegetable tanned leathers)
Sole leather	Apparent density, water absorption, resistance to abrasion, grain crackiness strength	Oils and fats, water solubles, ash, degree of tannage
Clothing leather	Tearing strength, stitch tear strength, shrinkage temperature, fastness to dry and wet rubbing as well as to washing and light, air permeability, water vapour permeability, cold crack resistance, drape	Oils and fats, water solubles, ash, Cr_2O_3 and other mineral matter, hide substance
Upholstery leather	Tensile strength, elongation, tearing strength, stitch tear strength, fastness to dry and wet rubbing	Oils and fats, water solubles, ash, Cr_2O_3
Leather for knee bushings	Shrinkage temperature, resistance to abrasion, water absorption	Oils and fats, water solubles, ash, hide substance, formaldehyde content
Belting leather	Tensile strength, elongation, grain crack strength, stitch tear strength	Oils and fats, water solubles, ash, hide substance, Cr_2O_3 (Epsom salt, glucose, acidity for vegetable beltings)

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Hydraulic leather	Shrinkage temperature, water absorption, grain crack strength, grain pipiness	Oils and fats, water solubles, ash, degree of tannage
Gasmeter diaphragms	Elongation, compressibility, resilience, area shrinkage, bursting strength	Oils and fats, water solubles, ash, Cr_2O_3
Chamois leather	Tensile strength, elongation, tearing strength, stitch tear strength, water absorption, sink test, petrol filtration	Oils and fats (free and fixed), ash
Leathers for football	Tensile strength, elongation (along different directions), stitch tear strength, grain crack strength, shrinkage temperature, water absorption	Oils and fats, water solubles, ash, Cr_2O_3 and other mineral matter, degree of tannage (for vegetable tanned leathers)
Book-binding leather	Tearing strength, stitch tear strength, resistance to perspiration, fastness to wet and dry rubbing, resistance to acid rotting	Oils and fats, water solubles, ash, hide substance
Snake skins	Breaking strength, elongation, stitch tear strength, tearing strength	Oils and fats, water solubles, ash, hide substance, Al_2O_3 (for aluminium-tanned leathers), degred of tannage (for vegetable-tannee leathers)
Miners' safety leather boots and shoes	Thickness of individual leather components and the upper, lining and sole (leathers) components conforming to the specifications of respective leathers, besides the specification for thread, steel toe cap, eyelet, etc.	
Safety leather boots for workers in heavy metal industries	As for the Miners' safety leather boots and shoes	
EI tanned leathers	Tensile strength, elongation, stitch tear strength, tearing strength, water absorption	Oils and fats, water solubles, ash, hide substance, leather substance (also tests for specific use such as linings, components for leather-goods, etc.)
Wet-blue leathers	Shrinkage temperature	Moisture, Cr_2O_3

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Leathers, in general, in their marketable form, must have pH values not less than 3.5 indicating the absence of free mineral acids and should have been treated with fungicides in optimal amounts as a protection against mildew growth during transit and storage; determinations of the pH values of water solubles and the difference figure are also obligatory for all types of leathers.

Actual quality requirements that are recommended for some of these leathers as well as for various other types of leathers are given in Appendix 1.

Details of the tests and the broad areas of application of these tests for different types of leathers are given in Chapter 3.
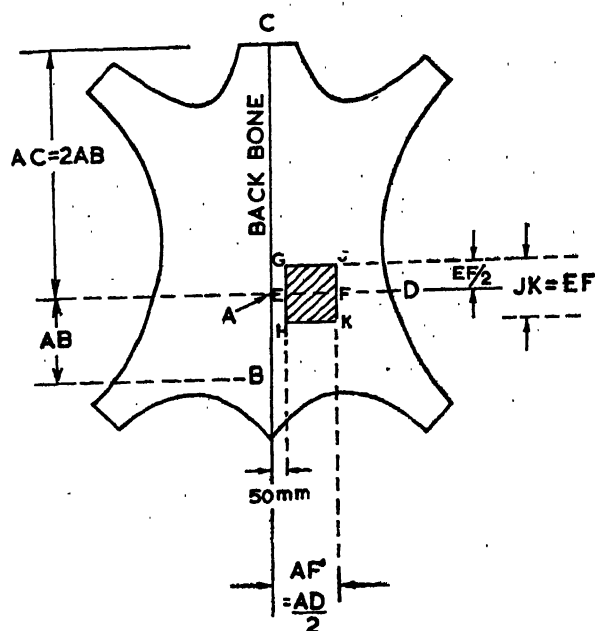
TESTING AND ANALYSIS OF LEATHERS

PHYSICAL TESTING OF LEATHERS

Leather finds place in consumers' articles in its natural form after being subjected to certain finishing treatments. These finishing treatments such as staining, polishing, etc., are done more for imparting an eye-appeal rather than for modifying the basic properties of the leathers themselves. The inherent properties of the leathers are predominant in the leatherware and are of utmost importance from the point of view of the consumers. The controls that are vital for maintaining the quality of the innumerable types of leathers that find use in a variety of human activities thus acquire a special significance as unquestionable pre-requisites in the production of leathers.

The quality of the final leather which is of great relevance to the consumer is the ultimate responsibility of the tanner, although many controls and changes are effected during the production-proper. The determination of physical and chemical characteristics of the leathers which indicate the quality of leathers is dealt with in this Chapter. Of the various physical characteristics of leathers namely, strength, elongation, porosity, absorption, flexural endurance, compressibility and resilience, fastness to light, weather, etc., only a combination of two or more properties is to be tested to ensure the quality of the leather for a particular use. For example, as indicated in Chapter 2, sole leathers are to be tested for water absorption as well as resistance to abrasion; upper leathers for flexural endurance, fastness to rubbing, water vapour permeability, etc.; belting leathers for tensile strength and elongation. The physical and mechanical properties to be determined on a given sample of leather must have relevance to its ultimate end-use.

Since leathers are made from natural raw material—hides and skins—they are not topographically uniform. Thus, for example, leather from the region near-about the backbone is very compact with the fibre-weave quite different as compared to leather from other areas. The leather from belly area is much looser and thinner. To ensure greater uniformity in the assessment of the quality of leathers, an agreed sampling position is followed in all the leather-testing laboratories of the world. Figures 1-4 indicate the sampling positions in respect of whole hides, sides and skins, bends, shoulders, and bellies.

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The specimens for physical tests in the case of whole hides, sides and skins (Figure I) are taken from the square HKJG and as close to the line EF as possible. B is the root of the tail. A is a point on the back bone such that AC=2AB. AD is a line perpendicular to BC. F is the midpoint of AD. AE is of 50 mm length. GH and JK are equal in length to EF and parallel to BC; E and F are their respective midpoints.
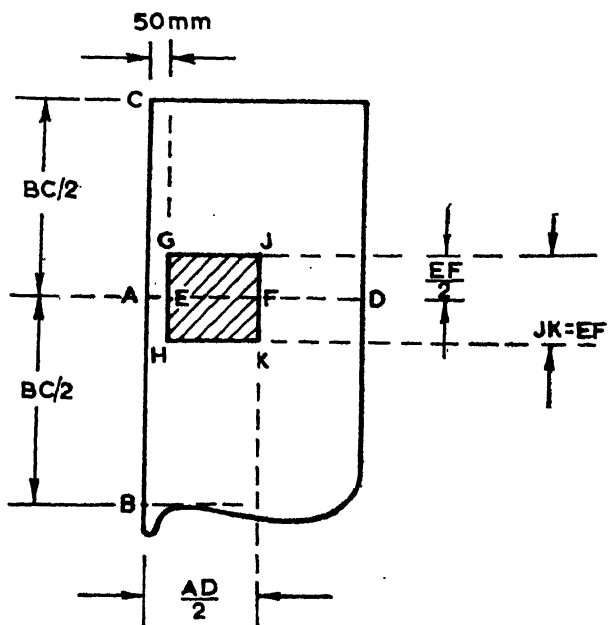

FIG. 1.

The test specimens for physical tests in the case of bends (Figure 2) are taken from the square HKJG and as close to the EF as possible. B is the root of the tail. A is a point on the backbone BC such that AC=AB. AD is perpendicular to BC. F is the midpoint of AD. AE is of 50 mm length. GH and JK are the lines equal in length to EF and parallel to BC; E and F are their respective midpoints.

The test specimens for physical tests in the case of shoulders (Figure 3) are taken from the rectangle ABCD and as close to C as possible. P is the midpoint of RS. DC is parallel to and 20 mm distant from RS and BC is parallel to the backbone. DA is parallel to and 50 mm distant from the backbone and of length equal to half the length of DC. AB is parallel to DC.

The test specimens for physical tests in the case of bellies (Figure 4) are taken from locations as close to the point Q as possible, but not less than 20 mm from the line TU. Q is the midpoint of the line TU.

14

50mm

BC/2

BC/2

C

G   J

A — E   F   D

H   K

B

$\dfrac{AD}{2}$

$\dfrac{EF}{2}$

JK=EF

Sampling position
of bends (butts)

FIG. 2.

50mm

$\dfrac{DC}{2}$

A   B

D   C

R   P   S

T

20mm

$\dfrac{RS}{2}$ $\dfrac{RS}{2}$

FIG. 3

Sampling position
of shoulders

SHOULDER

20mm

R   P   S

T

Q

BELLY
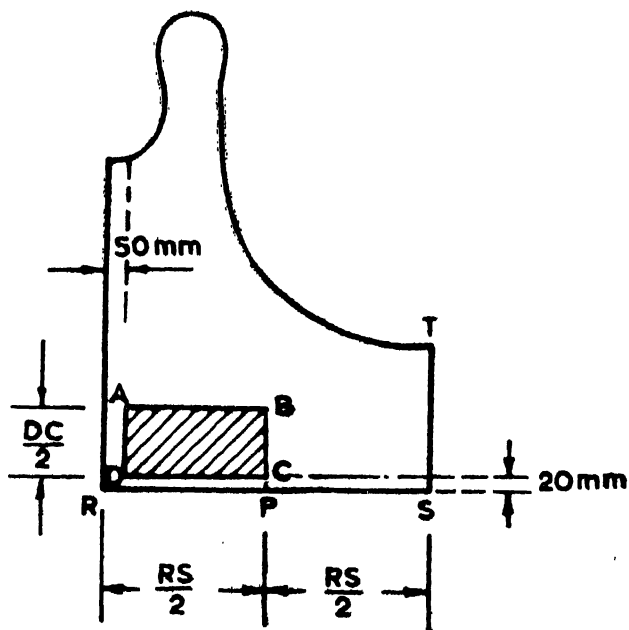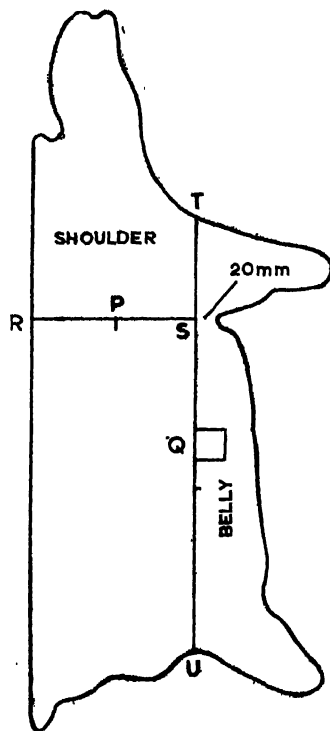
U

FIG. 4.

Sampling position
of bellies (flanks)

Just as the sampling positions are prescribed, the atmospheric conditions during testing leathers are also prescribed to ensure greater uniformity in the assessment of leather quality. A temperature of 27 ± 2 °C and relative humidity of 65 ± 2 per cent are maintained for physical testing. In the following, official test procedures for the determination of various physical characteristics of leathers are described.

TENSILE STRENGTH (LP6/IS: 5914-1970; SLP6; IUP/6)*

Tensile strength is the force (kg) per unit area of cross-section (sq.cm) required to cause a rupture of the test specimen.

The leather sample is cut into dump-bell (Figures 5-7) shapes (190- × 40 mm with 100 × 20 mm constriction in the middle; 90 × 20 mm with 50 × 10 mm constriction;

Leather specimen for determining tensile strength

FIG.5.

Leather specimen for determining tensile strength
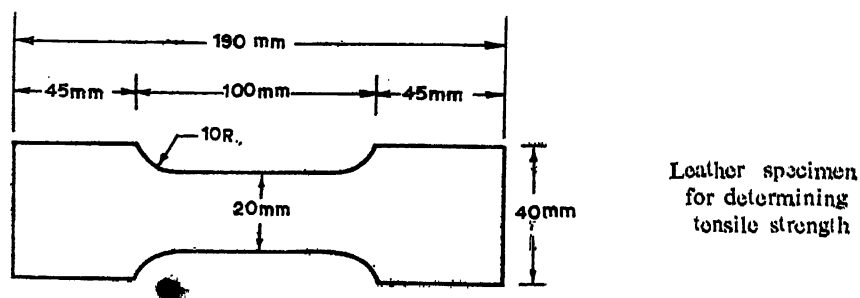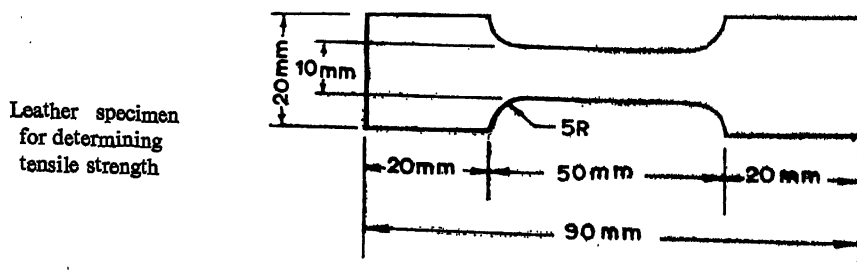
FIG. 6.

*LP—Methods of physical testing of leather.

IS—Indian standard published by the Indian Standards Institution, New Delhi.

SLP—Section: Physical testing of leather, published by the Society of Leather Trades, Chemists, UK.

IUP—Physical testing of leather, published by the International Union of Leather Chemists' Society.
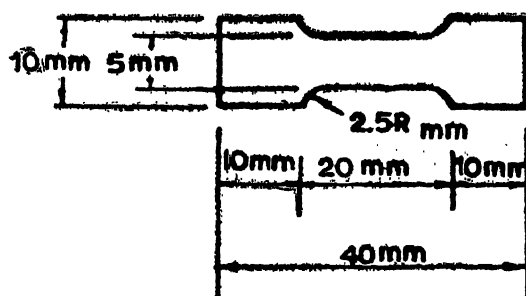
16

FIG. 7.

Leather specimen for determining
tensile strength

40×10 mm with 20×5 mm constriction) from the sampling position, both parallel and perpendicular to the backbone. The force of pulling gets concentrated at the narrow middle portion and the breaking takes place at this region. The thickness and the width of the sample are measured at a minimum of three points over the narrow middle portion and the respective averages calculated. The leather specimen is then clamped to the dynamometer (Figure 8). The speed of separation of the jaws is kept at 100±20 mm/minute. The load at which the leather breaks is noted.

$$\text{Tensile strength, kg/sq cm.} = \frac{\text{Breaking load (kg)}}{\text{Thickness (cm)} \times \text{Width (cm)}}$$

Application: As an index of quality of all types of leathers, and especially of belting, picking band and harness leathers.

TEARING STRENGTH (LP7/IS : 5914-1970; SLP 7; IUP 8)

Tearing strength is the load in kg required to tear the sample of leather of unit thickness (cm).

Leather specimens (50×25 mm) provided with a slot having the specified dimensions (Figure 9) or a straight cut of 20 mm length (Figure 10) for light leathers are used for determining tearing strength.

Sampling is done both parallel and perpendicular to the backbone and the mean thickness of the test specimens determined. Tearing is accomplished at the dynamometer using a test piece holder (Figure 11) and the maximum recorded load is noted.

$$\text{Tearing strength, kg/cm thickness} = \frac{\text{Tearing load (kg)}}{\text{Thickness (cm)}}$$

Application: All leathers meant for the fabrication of footwear and leather-goods (e.g., upper leather, clothing leather, gloving leather, etc.).
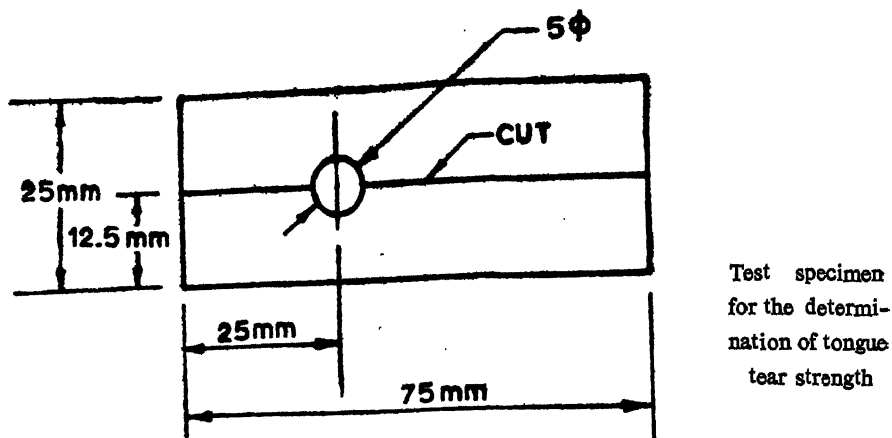
17

T—3

FIG.12.

Test specimen for the determination of tongue tear strength

STITCH TEAR STRENGTH (DOUBLE HOLE) (LP8/IS:5914-1970; E 13/ ALCA*)

The load (kg) required to tear the sample of leatherbetween two holes of 2 mm diameter each and whose centres are 6 mm apart, expressed on its unit thichness (cm) is the stitch tear strength.

The test specimen for this test is of the dimensions shown in Figure 13. The sampling is carried out in both parallel and perpendicular directions to the backbone and the mean thickness is determined at the area of the leather of 6mm length throughout its 25 mm width. A soft steel wire (of 1 mm diameter) of about 10 mm length bent into U-shape, is introduced into the holes of the test piece from the grain side in such a way that the two ends of the wire project at the flesh side. The wire is wrapped and then clamped
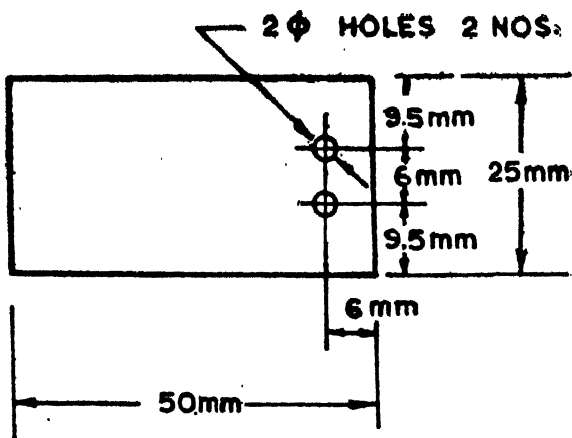

FIG.13

Test specimen for the determination of stitch tear strength

* ALCA—American Leather Chemists' Association, USA.

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to the upper jaws of the dynamometer; the test specimen is fixed to the lower jaws. The tearing load is determined.

$$\text{Stitch tear strength (double hole), kg/cm thickness} = \frac{\text{Tearing load (kg)}}{\text{Thickness (cm)}}$$

In the case of heavy leathers, only one hole and a steel rod instead of the two holes and the steel wire are made use of.

Application: All leathers meant for the fabrication of footwear and leather goods.

BUCKLE TEAR STRENGTH

The load (kg) required to tear a test specimen of leather joined to another piece of test specimen with a buckle, expressed per unit thickness (cm) is the buckle tear strength.

A convenient size of a pair of test specimens joined with a buckle of convenient dimensions is used for this test. Tearing is done in the dynamometer and the tearing load determined. The dimensions of the tongue of the buckle is indicated in the test report along with the value of the buckle tear strength.

Application: Leathers used as straps with buckles.

SPLIT TEAR STRENGTH

The load in kg required to continue the split in the leather specimen, expressed on its unit thickness (cm) is the split tear strength.

This test is conducted as for tongue tear strength, excepting that the cut is made at as near as possible to the middle of the thickness of the test specimen, i.e., parallel to the surface, instead of perpendicular to the surface.

Application: To assess the quality of the leathers in general.

ELONGATION AT BREAK (LP 6/IS: 5914—1970; SLP 6; IUP 6)

The extent of elongation of the leather specimen at the time of its breaking, while applying the tensile force, expressed as the percentage on the original length of the said specimen is the elongation at break.

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This characteristic is found during the determination of tensile strength. The distances between the two separating jaws of the dynamometer before starting the instrument (i) and at the time of breaking of the test specimen (f) are noted. The difference between the two (f — i), expressed as a percentage of (i) is the percentage elongation at break.

$$\text{Elongation at break, per cent} = \frac{(\text{Final distance between the jaws} - \text{Initial distance between the jaws})}{(\text{Initial distance between the jaws})} \times 100$$

This test provides information as to whether a particular sample of leather is brittle (very less value) or weak and elastic (very high value). Brittleness, weakness, elasticity, etc., are confirmed through other strength characteristics as well. It is to be noted however, that the nature and extent of a tannage confer a high or low elongation, e.g., a normal chrome tannage produces leathers with greater elongation than a normal vegetable tannage does.

Application: For all types of leathers, especially for beltings, picking band and harness leathers.

ELONGATION AT A SPECIFIED LOAD (LP6/IS: 5914—1970; SLP 6; IUP6)

The extent of elongation of a leather specimen when subjected to a specified tensile load, expressed as the percentage on the original length of the said specimen is the elongation at that specified load.

The area of cross-section (a sq. mm) of the narrow middle portion of the dumb-bell shaped test specimen is first calculated through mean thickness (mm) and mean width (mm). The product of area of cross-section (a sq. mm) and the specified tensile force (e.g., 1 kg/sq. mm, 2 kg/sq. mm) gives the load in kg (1a; 2a) at which the elongation is to be determined. The test piece is clamped on the dynamometer, two reference marks at convenient distance apart (l_0) are marked in the narrow middle portion of the test specimen and the load (kg) arrived at is applied. The distance (l_1) between the two reference marks at the end of 15 minutes, when the load is acting, is measured. The difference between l_1 and l_0, expressed as a percentage of l_0 is the per cent temporary elongation.

$$\text{Temporary elongation at } - - \text{ kg/sq. mm, per cent} = \frac{l_1 - l_0}{l_0} \times 100$$

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The test specimen is taken out of the dynamometer, allowed to rest for a given time (usually 4 hours) and the distance between the reference marks measured (l_2)

$$\text{Permanent elogation at} - \text{kg./sq. mm., per cent} = \frac{(l_2 - l_0)}{l_0} \times 100$$

Application: Belting, picking band, harness, football, etc., leathers.

GRAIN CRACK INDEX (LP13/IS: 5914—1970; SLP8; IUP 12)

The grain crack index is the product of the number (n) of the largest mandrel, bending (grain outwards) around which the grain of the leather cracks, and the thickness (tmm.), of the test specimen (Table 2).

TABLE 2

Mandrel Number (n)	Mandrel Diameter (mm)
1	61.67 ± 0.03
2	35.00 ± 0.03
3	23.57 ± 0.03
4	17.22 ± 0.03
5	13.18 ± 0.03
6	10.38 ± 0.03
7	8.33 ± 0.03
8	6.76 ± 0.03

Application: All leathers other than suedes and chamois. The greater the value for grain crack index, the better the resistance of the grain to cracking.

EXTENSION OF THE GRAIN AT GRAIN CRACK (LP 13/IS: 5914—1970; SLP 8; IUP 12)

The extension of the grain caused by bending the leather around a series of mandrels of varying diameters, grain outwards, is read as a percentage from Table 3.

TABLE 3

GRAIN CRACK INDEX(a) AND PER CENT EXTENSION OF THE GRAIN AT GRAIN CRACK (b)

Mandrel Number (n)	Thickness of leather in mm.									
	3		4		5		6		7		8	
	a	b	a	b	a	b	a	b	a	b	a	b
1	3	—	4	—	5	—	6	—	7	—	8	—
2	6	6	8	8	10	10	12	12	14	13	16	15
3	9	10	12	12	15	15	18	17	21	20	24	22
4	12	13	16	17	20	20	24	23	28	26	32	29
5	15	17	20	21	25	25	30	29	35	32	40	35
6	18	20	24	26	30	30	36	34	42	37	48	41
7	21	24	28	30	35	35	42	39	49	43	56	46
8	24	29	32	35	04	40	48	44	56	48	64	52

For a leather of 5 mm thickness, the grain crack index (nt) is nearly equal to the percentage extension of the grain at grain crack.

Application: All leathers other than suedes and chamois.

GRAIN CRACK STRENGTH (SLP 9; IUP 9)

The pressure required to cause first signs of crackiness on the grain is the grain crack strength.

The pressure is accomplished either by hydr. ulic means (glycerine or any non-compressible liquid) through a rubber diaphram as in the Mullen apparatus (Figure 14) or by mechanical means through a spherical head as in the lastometer. A circular test specimen is used and the thickness is read at the middle of the specime. before starting the test.

Application: All leathers with grain layer. This test gives information on the degree of resistance the grain offers to the pressures to which the leather is subjected to.
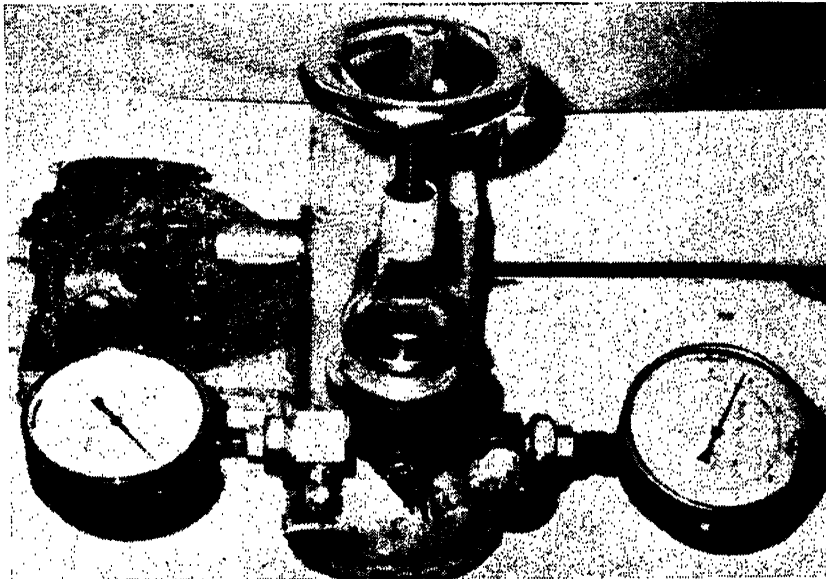
Fig. 14

Mullen apparatus

BURSTING STRENGTH (SLP 9; IUP 9)

Bursting strength is the pressure required to burst the leather.

The bursting strength is determined by continuing the pressure beyond the grain crackiness till the leather bursts.

Application: Oil seal leather and hydraulic (washer) leather. The bursting strength is an index of the overall strength of the leathers and is generally determined along with the determination of grain crack strength.

TWO-DIMENSIONAL EXTENSION (LP 14/ IS: 5914-1970; SLP10; IUP13)

The per cent area extension (A) and the per cent radial extension (R) in addition to the linear tension, kg/cm (T) of the leather at (1) the first crack of finish, (2) the first crack of the grain, and (3) the burst of the leather are determined by using the tensometer (Figure 15).
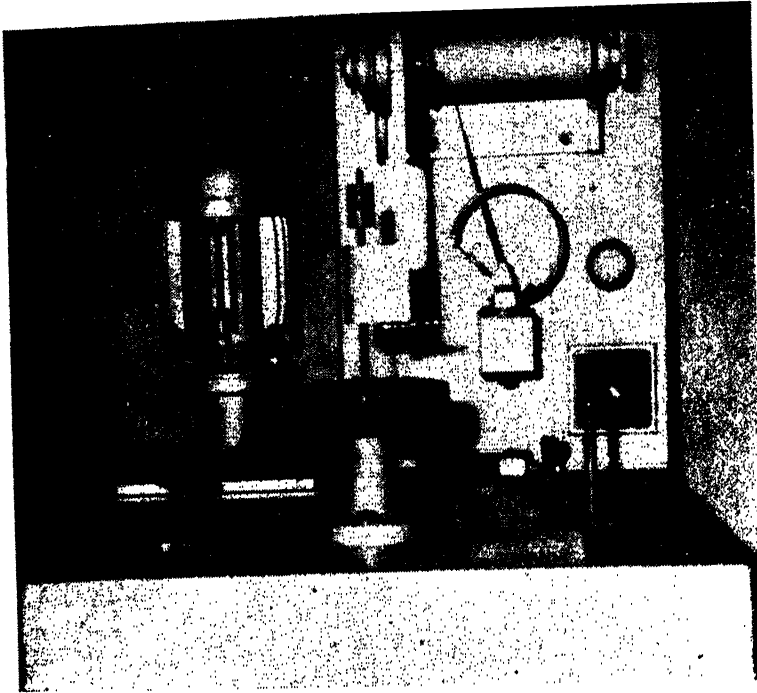
T—4

Fig. 15

Tensometer

Hydraulic pressure is applied to the circular leather specimen through a rubber diaphram as in the Mullen apparatus. The tensometer, in addition, is provided with an auto-recorder for graphically recording the pressures in kg/cm^2 (ordinates), and the heights (h) of the cap in mm (abscissae).

For a given pressure (pressure required to cause the first crack of the finish, the first crack of the grain and, the burst of the leather), h is read from this plot and from its value the per cent area extension (through the formula $A = 0.16 \, h^2$) and the percent radial extension (from Table 4) are obtained. The linear tension, (kg/cm) is given by the formula $T = pZ$, where p is the force (kg/cm^2) and the values of Z are read from Table 4. The values of area extension, radial extension, and linear tension can be found out for pressures of 5, 10, and 15 kg/cm^2 while ascending as well as descending.

Application: All types of finished light leathers.

TABLE 4

VALUES OF PERCENTAGE RADIAL EXTENSION (R) AND z FOR VARIOUS VALUES
OF CAP HEIGHT (h) IN mm

h	R	Z	h	R	Z
0.25	0.01	62.51	7.25	5.5	2.34
0.50	0.04	31.26	7.50	5.9	2.27
0.75	0.07	20.85	7.75	6.3	2.21
1.00	0.11	15.65	8.00	6.7	2.15
1.25	0.17	12.53	8.25	7.1	2.10
1.50	0.24	10.45	8.50	7.5	2.05
1.75	0.33	8.97	8.75	8.0	2.00
2.00	0.43	7.86	9.00	8.4	1.96
2.25	0·54	7.00	9.25	8.9	1.92
2.50	0.66	6.31	9.50	9.4	1.88
2.75	0.80	5.75	9.75	9,9	1.85
3.00	0.96	5.28	10.00	10.3	1.81
3.25	1.1	4.89	10.25	10.9	1.781
3.50	1.3	4.55	10.50	11.4	1.751
3.75	1.5	4.26	10.75	11.9	1.722
4·00	1.7	4.01	11.00	12.5	1.695
4·25	1.9	3.78	11.25	13.0	1.670
4.50	2.1	3.58	11.50	13.6	1.646
4.75	2.4	3.41	11.75	14.1	1.624
5.00	2.6	3.25	12.00	14·7	1·602
5.25	2.9	3.11	12.25	15.3	1.582
5.50	3.2	2.98	12.50	15.9	1.563
5.75	3.5	2.86	12.75	16.5	1.544
6.00	3.8	2.75	13.00	17.2	1.527
6.25	4.1	2.66	13.25	17.8	1.510
6.50	4.5	2.57	13.50	18.4	1.495
6.75	4.8	2.48	13.75	19.1	1.480
7.00	5.1	2.41	14.00	19.7	1.466

TABLE 4 (Contd.)

h	R	Z	h	R	Z
14.25	20.4	1.453	19.75	37.5	1.285
14.50	21.1	1.440	20.00	38.3	1·281
14.75	21.8	1.428	20.25	39.2	1.278
15.00	22.5	1.417	20.50	40.1	1.275
15.25	23.2	1.406	20.75	41.0	1.271
15.50	23.9	1.396	21.00	41.9	1.269
15.75	24.6	1.386	21.25	42.8	1.267
16.00	25.4	1.377	21.50	43·7	1.264
16.25	26.1	1.368	21.75	44.6	1.262
16.50	26.9	1.359	22.00	45.5	1.260
16.75	27.7	1.352	22.25	46.4	1.258
17.00	28.4	1.344	22.50	47.4	1.257
17.25	29.2	1.337	22.75	48.3	1.256
17.50	30.0	1.330	23.00	49.3	1.254
17.75	30.8	1.324	23.25	50.2	1.253
18.00	31·6	1.318	23.50	51.2	1.252
18.25	32.4	1.312	23.75	52.2	1.252
18.50	33.2	1.307	24.00	53.1	1.251
18.75	34.1	1.302	24.25	54.1	1.251
19.00	34.9	1.297	24.50	55.1	1.250
19.25	35.8	1.293	24.75	56.1	1.250
19.50	36.6	1.289	25.00	57.1	1.250

GRAIN PIPINESS (LP 22/IS: 582—1954)

When a leather is bent through 180° over a mandrel of given diameter, grain side inwards, there is a tendency of piping of the grain layer as distinct from the corium. The extent of such piping is viewed along the edges after removing the mandrel and is a measure of the looseness of the grain layer. The test is usually conducted over a series of mandrels of decreasing diameters.

Application: Mostly heavy leathers.

The inverse of loss in thickness of leather due to an abrasive action is its resistance to such abrasion. Different countries have different systems to produce the abrasive action for testing abrasive resistance. In the present circumstances, this test can at best serve for in-plant lot-to-lot comparison.

While heavy leathers can be tested by a rubber abrader (abrasive medium: paper) (Figure 16), a Taber abraser (abrasive medium: stone) (Figure 17) can be used for light leathers. The loss in thickness after a given number of rotations of the abrading drum or leather mounted disc is measured using micrometer or thickness gauge.
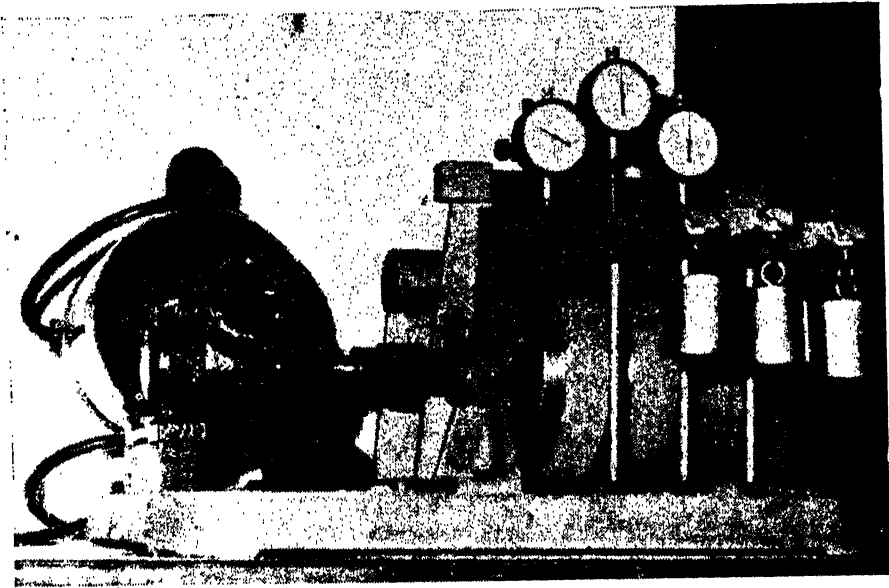

Fig. 16

Rubber abrader

WATER ABSORPTION (STATIC) (LP12/IS: 5914—1970; SLP19; IUP7)

The amount of water absorbed by a given weight of leather in a given time under stationary condition, expressed as a percentage on the weight of the leather, is the per cent water absorption of the leather in that given time.

This test is usually conducted for 30 minutes, 2 hours and 24 hours' contact of the test specimen with water. Circular test specimen and Kubelka apparatus
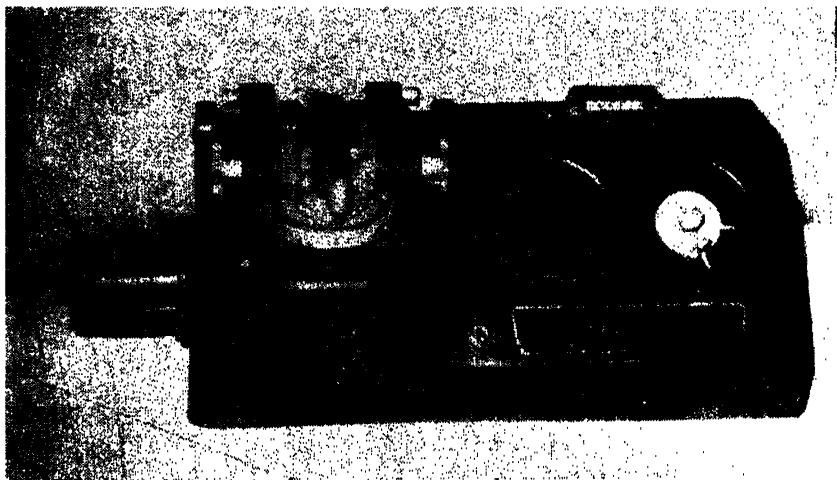
Fig. 17

Taber abraser

(Figure 18) are used for this purpose. Generally, the volume (ml) of water absorbed (which is roughly equivalent to the weight in g) is measured and is taken as the basis for calculating the per cent water absorption.

Fig. 18

Kubelka apparatus

$$\text{Water absorption in time t, per cent} = \frac{\text{Volume (ml) of water absorbed in time t}}{\text{Weight (g) of leather specimen}} \times 100$$

In certain cases (LP11/IS: 5914—1970; SLP 20), the weight of the test specimen is determined at the end of time t after lightly blotting it with a dry blotting paper and the percentage water absorption is calculated with the increase in the weight of the test specimen in the given time t as the basis. In either case—decrease in the volume of water or increase in the weight of leather—no correction is generally given to the removal of water soluble matter from the leather.

Application: Sole leather.

WATER PENETRATION (STATIC)

The time taken by a column of water to penetrate through the leather thickness (grain to flesh) is measured as an indication of static water penetration.

Fifty millilitres of water are taken in the top cylinder (50 mm dia.) of an upright glass cylinder-leather-glass cylinder assembly and the time taken for the first drop of water to penetrate through the leather is recorded. The leather-glass cylinders contact points are sealed with molten paraffin wax along the edges.

Application: Sole leather.

WATER PROOFNESS OF SOLE LEATHERS (DYNAMIC) (LP18/IS: 5914—1970; SLP 23; IUP 11)

The time taken for water to penetrate through the entire thickness of the leather, the gain in weight (i.e., water absorption of the test specimen in a given time), and the rate of penetration of water are determined while the leather is being flexed and compressed simulating the sole of a shoe while walking, using permeometer (Figure 19) in this test.

Leather specimens for this test are of dimensions 100 × 40 mm. The size of the absorbent board for measuring the rate of penetration of water is 100 × 60 mm. In general, water absorption and water penetration rates are measured at intervals of 1 hour (e.g., 1, 2, 3,. . . ., hours') flexing. The water absorption is finally expressed as the percentage on the original weight of leather. The rate of water penetration is determined during a period of 10 minutes and is expressed as g/dm^2/h.
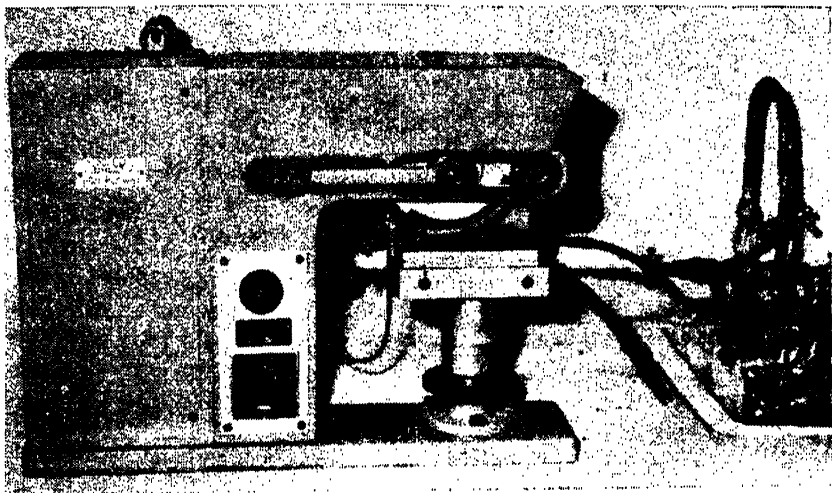
Application: Sole leather.

Fig. 19

Permeometer

WATER PROOFNESS OF UPPER LEATHERS (DYNAMIC) (LP 20/ IS: 5914-1970; SLP 22; IUP 10)

The penetration time, the water absorption in a given time, and the amount of water transmitted during a given time interval are determined while the leather is being flexed simulating the conditions of wear, using penetrometer (Figure 20) to obtain an indication of water proofness.

The test piece is of size 75×60 mm. The size of the absorbent cloth (e.g., terry towel) is 120×40 mm, weighing about 300 g/m². Water absorption is measured as the difference in the weights of leather-cylinder assembly at hourly intervals, from the beginning of the test, and is expressed as the percentage on the original weight of the leather. The amount of water transmitted at a given time interval is measured as the difference in the weights of the absorbent cloth, at hourly intervals, from the beginning of the first hour after the occurrence of penetration and is expressed as g.

Application: Upper leather.

AIR PERMEABILITY

The time taken for a given volume of air under a given excess pressure to pass through a fixed area of the leather till the pressure becomes equal to that of the atmo-
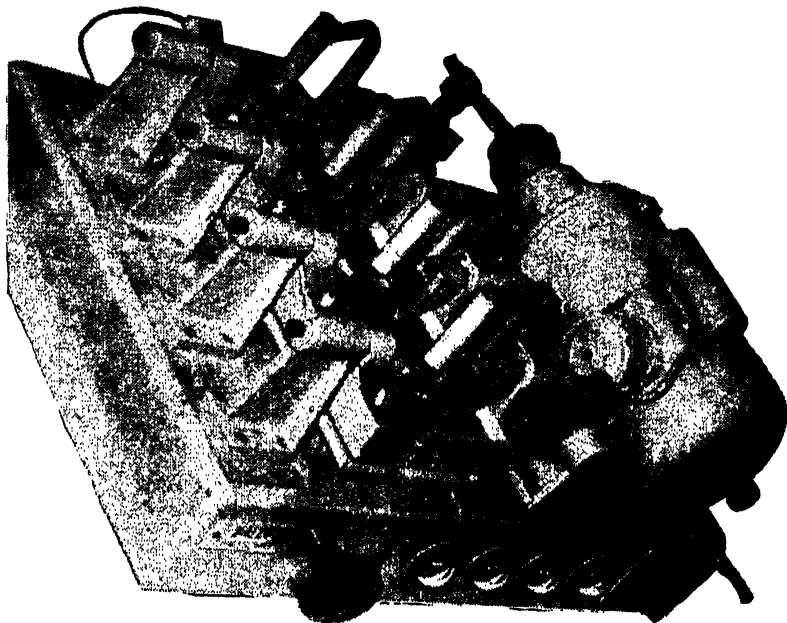
32

Fig. 20

Penetrometer

sphere (Figure 21) is noted. The porosity of leather to air is thus determined. Better porosity (less time) provides better comfort to the wearer when such leathers are used as clothing. Insignificant or lesser porosity is a pre-requisite for washer and seal leathers.

Application: Clothing leather, gloving leather, upper leather, washer leather and seal leather.

WATER VAPOUR PERMEABILITY (LP21/ IS: 5914—1970; SLP 25; IUP 15)

The water vapour permeability is the amount (mg) of water vapour passing through unit area (cm^2) of the leather specimen in a unit time (h).

The leather specimen separates the conditioned atmosphere and the solid desiccant (dry silica gel). The bottle containing the desiccant and covered with the test specimen is kept in a rapid current of air in the conditioned room and the air within the bottle is also circulated by keeping the desiccant in motion (Figure 22). This is a very important physical characteristic of leathers for assessing their comfort properties.

Application: Clothing leather, gloving leather and upper leather.

33

T—5

Fig. 21

Air permeability
apparatus

APPARENT DENSITY (LP5/IS: 5914—1970; SLP5; IUP5)

The apparent density of a sample of leather is its mass per unit overall volume.

For the determination of the apparent density, a 70-mm diameter circular leather specimen is weighed (g). The thickness in cm (t) and hence the volume are then determined.

$$\text{Apparent density} = \frac{\text{Weight in g}}{\text{Volume in cc}}$$

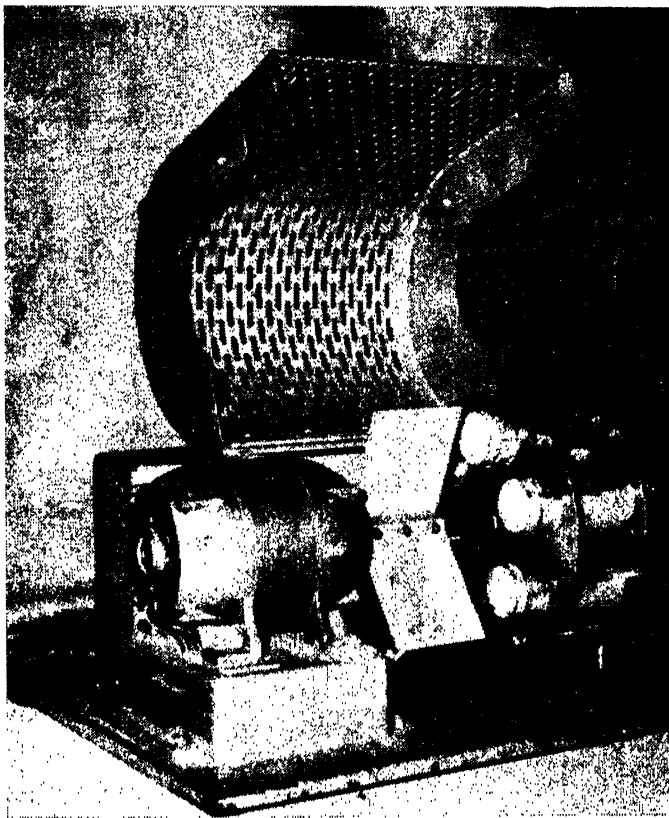*Application:* Sole leather and other heavy leathers.

34

Fig. 22

Water vapour permeability apparatus

REAL DENSITY

The real density of a sample of leather takes into account the volume of its tanned fibres.

The mass m of the test specimen is easily determined by weighing while the real volume (i.e., volume of the tanned fibres) is determined by gas displacement method. For this the air-dry leather is placed in a bulb of volume v_1 under pressure p_1; the volume occupied by the air and leather gets reduced to v_2 when the pressure is increased to p_2. The real volume v of the leather is given by the expression $(p_2 v_2 - p_1 v_1)/(p_2 - p_1)$ as per the Boyle's law. The real density of the leather (m/v) is thus obtained. A rough estimation of real density is possible by dividing the weight of the leather in air with the product of weight in an inert liquid (e.g., kerosene) and the density of the liquid.

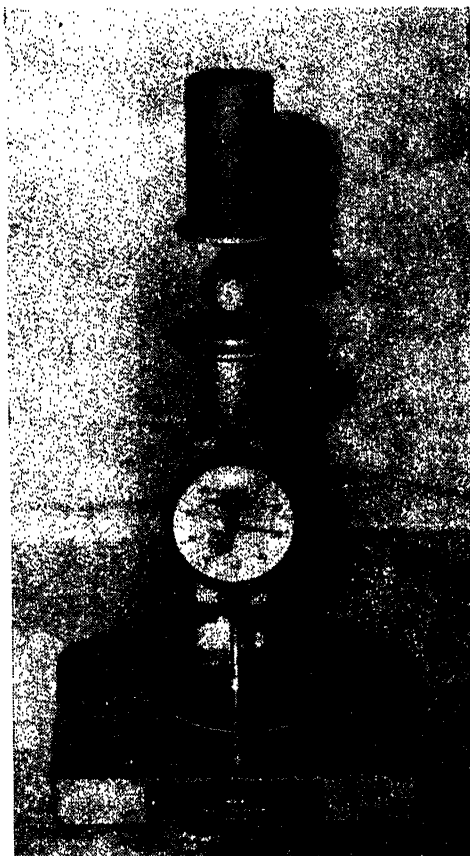*Application:* General information on all types of leathers.

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Compressibility is the extent to which the leather gets compressed in its thickness under a given load and is expressed as a percentage over its original thickness. Resilience is the rebound thickness after removing the said load and is expressed as a percentage over the extent of compression.

These are determined with the Compressibility and Resilience (C & R) tester (Figure 23) which in principle, is a micrometer with a flat presser foot that rests on the test specimen.

Fig. 23
Compressibility and Resilience (C&R) tester

$$\text{Compressibility, per cent} = \frac{(\text{Original thickness} - \text{Compressed thickness})}{\text{Original thickness}} \times 100$$

$$\text{Resilience per cent} = \frac{(\text{Recovered thickness} - \text{Compressed thickness})}{(\text{Original thickness} - \text{Compressed thickness})} \times 100$$

Application: Roller leather and apron leather.

COEFFICIENT OF FRICTION

The friction offered by the grain surface of the test specimen against a smooth glass plate is determined by noting the minimum load required to just start the movement of the test specimen over the glass plate, the grain side facing the glass.

Application: Roller leather and apron leather.

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DRAPE COEFFICIENT

The draping quality of clothing leathers is determined by using a drapemeter (Figure 24). The test specimen is held by a supporting disc under a light source when the shadow falls on a photosensitive blue print paper which is then developed in an atmosphere of ammoniacal vapours. The ratio of the projected area under the draped specimen to its theoretical maximum is the drape coefficient.

Application: Clothing leathers.

SHRINKAGE TEMPERATURE
(LP 10/ IS: 5914—1970; SLP 18; IUP 16)

The temperature at which the leather just starts shrinking when heated in a liquid-medium is its shrinkage temperature or hydrothermal stability in that liquid. This test gives an idea of the extent of tanning action, i. e., cross-linking within the leather matrix.

Fig. 24
Drapemeter

The shrinkage tester (Figure 25) consists essentially of a heater, stirrer and a thermometer. The medium is generally water. If the shrinkage temperature of the leather is beyond 100°C, a mixture of glycerine and water in the ratio of 3:1 or liquid paraffin is used in the place of water. The rate of heating is 2°C per minute.

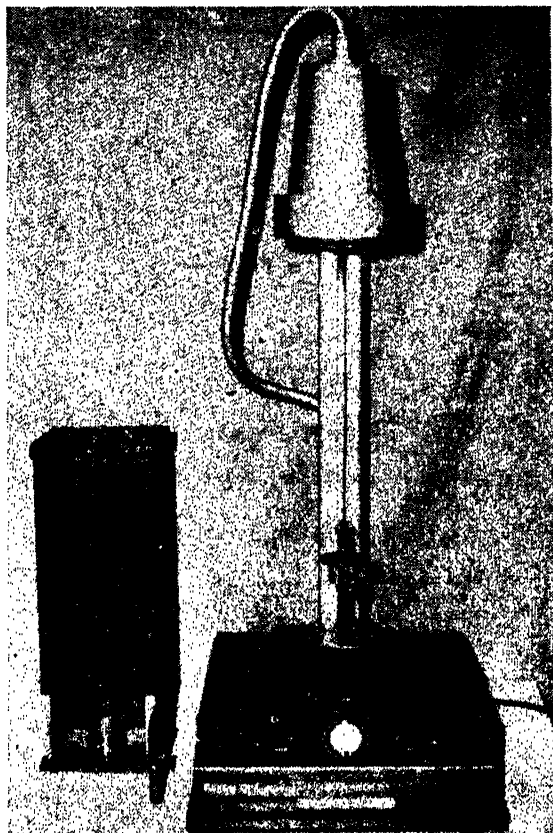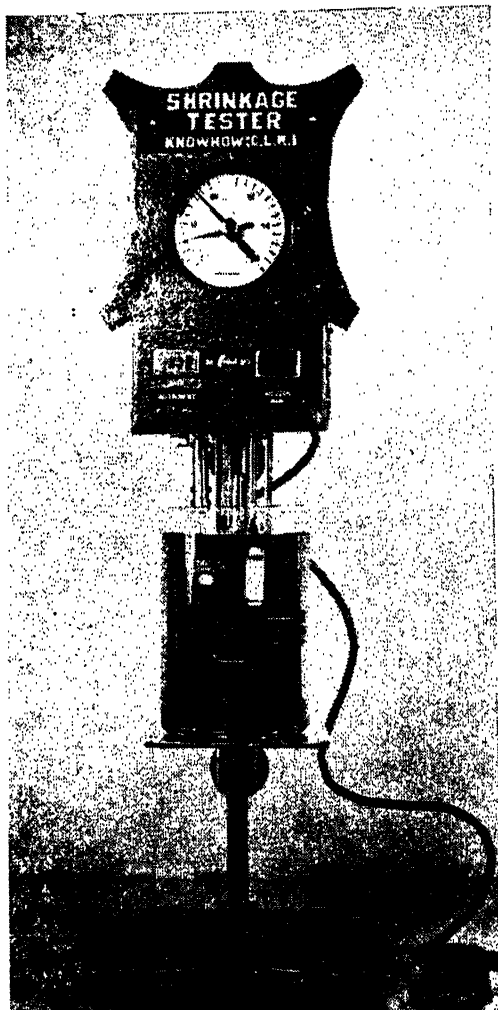*Application:* All types of leathers.

Fig. 25

Shrinkage tester

This test is intended to assess the flexing endurance of the finish-layer of any type of light substance finished leather. Flexing is accomplished in folded condition of the leather with a Bally flexometer (Figure 26). The number of flexes at which first signs of crack of finish layer appear is noted by periodically observing the test specimen. If the test is allowed to continue, the crackiness of the finish layer would be followed with the crackiness of the grain and finally with the complete failure of the leather itself.

Application: Light substance finished leathers.

FASTNESS TO RUBBING (WET
AND DRY) (*LF9/IS: 6191-
—1971; SLF* 5)

This is a very useful test for finished leather. A revolving pad is made to rub the sleather (Figure 27) for a uccessive number of revolutions and the effects of such rubbing under both the wet and dry conditions are studied using a Grey scale. While a 2.5 kg. load is used for dry rubbing, a 730 g load is used for wet rubbing. The number of revolutions for dry rubbing are 32, 64, 128, 256, 512 and 1024 (or 2048) and for wet rubbing 8, 16, 32, 64, 128, 256, 512 and 1024, every time exposing a

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* LF — Colour Fastness Test Methods

* SLF — Section: Colour Fastness of Leather, published by the Society of Leather Trades' Chemists, UK.
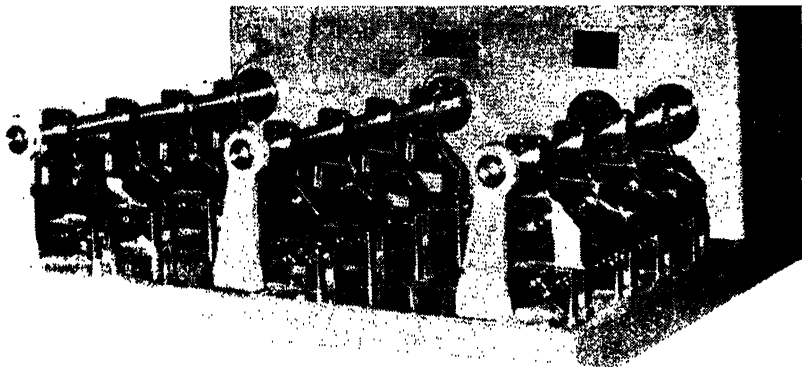
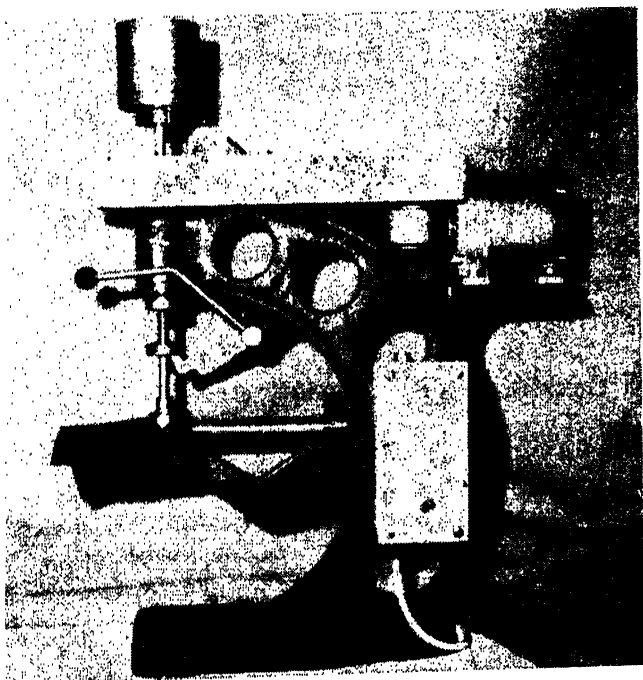38

Fig. 26

Bally flexometer

Fig. 27

Finish rub fastness tester

fresh surface of the leather for either type of rubbing. The number of revolutions required to cause a contrast of Grade 4, the number of revolutions required to cause a contrast of Grade 1 (if this is 1024 or less), and the contrast Grading after 1024 revolutions are found out and a description of the nature of such change is recorded.

Application: All types of finished leathers.

RESISTANCE AND FASTNESS TO PERSPIRATION (LF7/IS: 6191—1971; SLF 10)

The resistance of leathers to the action of artificial perspiration solution, with respect to their mechanical properties such as the loss of tensile strength, grain crackiness strength, etc., area loss, in general or in respect of staining of the adjacent material in the case of finished leathers, is an important physical characteristic for leathers such as clothing, lining and gloving.

The test specimen of required size is treated with the perspiration solution during a given time and after drying the treated leather at temperatures below 40°C, the losses in strength properties, hydrothermal stability, etc., are studied. In the case of assessing the fastness to perspiration, the test specimen (5×4 cm) is kept in between two pieces of undyed woollen textile (5×5 cm), a glass plate is placed on the composite specimen and is then brought in contact with the perspiration solution (1:50) for 30 minutes; the composite specimen with the glass plate is then maintained at $37 \pm 2°C$ for 4 hours, the leather piece is separated from the woollen cloth, dried in air, and the changes in colour on either side of the leather piece and the cloth are assessed with Grey scale and staining scale.

Application: Clothing leather, lining leather, etc.

FASTNESS TO LIGHT (LF3/4: IS: 6191—1971; SLF 1)

The fastness to light of dyed and/or finished leathers on exposure to light is an important characteristic and is accurately estimated by using a Xenotest (Figure 28) or similar instrument. The test specimen is exposed to Xenon lamp, under prescribed conditions, along with eight dyed-wool standards. The fastness of the test specimen is assessed by comparing the fading of the specimen with that of the standards. Any change in the colour of the test specimen is assessed by the Grey scale.

FASTNESS TO WEATHER

The behaviour of the leather when exposed to pre-set weather conditions (temperature, humidity, rain, etc.) is found out by using the Xenotest or similar instru-

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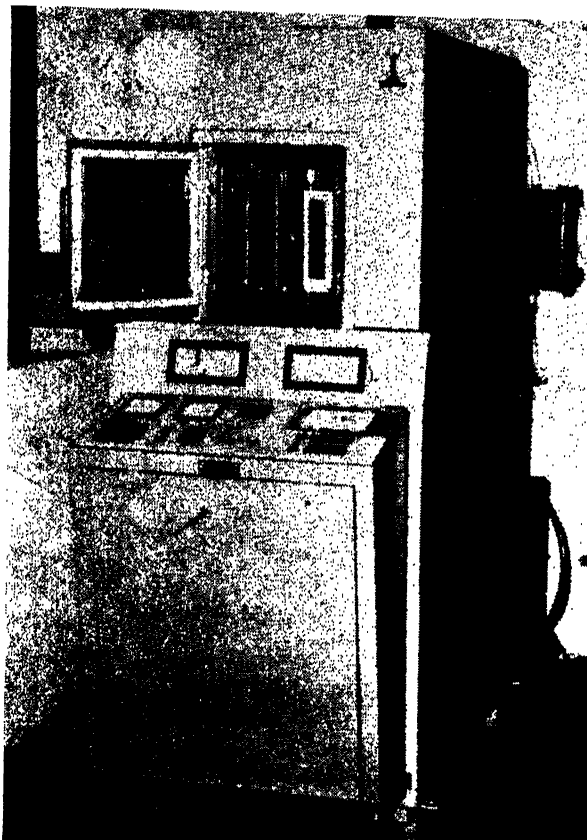
Fig. 28

Xenotest

ment.   The strength properties and other characteristics required of such treated
leathers are then examined and compared with those of unexposed controls.

RESISTANCE TO HEAT OF FINISH LAYER

This test gives information on the magnitude of resistance of the finish to heat and
is useful in cases where the finish layer is exposed to heat, as in the injection mould-
ing.   A band of the leather is brought into contact with a series of metallic plates
at increasing temperatures (Figure 29) for a given time and the behaviour of the
finish layer studied.   Beyond a particular plate, the finish layer is found to have

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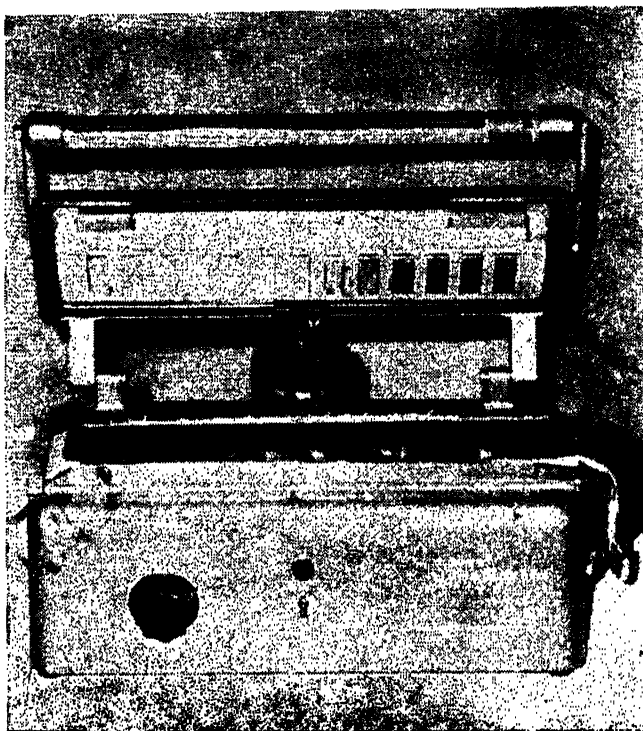
Fig. 29

Thermotest apparatus

deformed or melted and hence it is safer to use that leather at temperatures lower to the temperature of that particular plate.

Application: Finished upper leathers.

RESISTANCE TO COLD TEMPERATURE

The finish layers on the surface of the leather must be stable to colder temperatures at which they are required to be used. If not, they become brittle and crack on folding. The test specimen (75×75)mm of the finished leather is kept in the low temperature cabinet (generally at —20° C) for a desired length of time (usually 4 hours) and the finish layer then checked for crackiness,if any,on single folding within the cabinet itself. In the absence of crackiness on single folding, the finish layer is subjected to double folding and examined for crackiness. In addition to finish crackiness, the formation of fatty acid spues if any, on either side of the leathers. can also be examined.

Application: All types of finished leathers and leathers meant for use at colder temperatures.

RESISTANCE TO ACID-ROTTING

This test is intended to find out the resistance of leathers to acidified hydrogen peroxide and is called the Innes' peroxide test.

A 60×60 mm test specimen is treated with IN sulphuric acid (1 ml acid per g of the leather), air-dried for 24 hours, hydrogen peroxide (10 volumes) at the rate of 0.6 ml per g. of the leather added evenly, air-dried for 24 hours, the process of addition of hydrogen peroxide and the subsequent air-drying continued for a further period of six days, and the specimen assessed for crackiness and suppleness.

Application: Book-binding leathers.

CHEMICAL ANALYSIS OF LEATHERS

The chemical characteristics of leathers indicate the nature and proportion of components with which they are made of. One can thus find out by determining the chemical characteristics whether a particular sample is a normal leather or made up of ingredients in abnormal (or sub-normal) proportions. Chemical analysis of leather helps in finding the nature and extent of adulteration, if any.

Product and process controls during the manufacturing enable to produce the desired qualities in the final leathers which are, in turn, subjected to such analysis to check for their ultimate characteristics. Each chemical characteristic has definite influence on the quality of the final leather and deserves to be estimated towards obtaining an overall picture about the test specimen.

As in the case of physical testing, sampling position for chemical analysis is specified by official specifications. For in-plant control purposes, however, the procedure of edge-sampling can be adopted. The sampling positions in respect of whole hides, sides and skins, bends, shoulders and bellies are indicated in Figures 30—33.

For sampling whole hides, sides and skins, a square piece of leather (Figure 30) adjacent to the line HK, in the direction of the root of the tail, is taken from the joint H and with the side length equal to the half of HK. If necessary, the section can also be taken from the corresponding place in the opposite half of the leather· Figure 30 may be studied along with Figure 1.
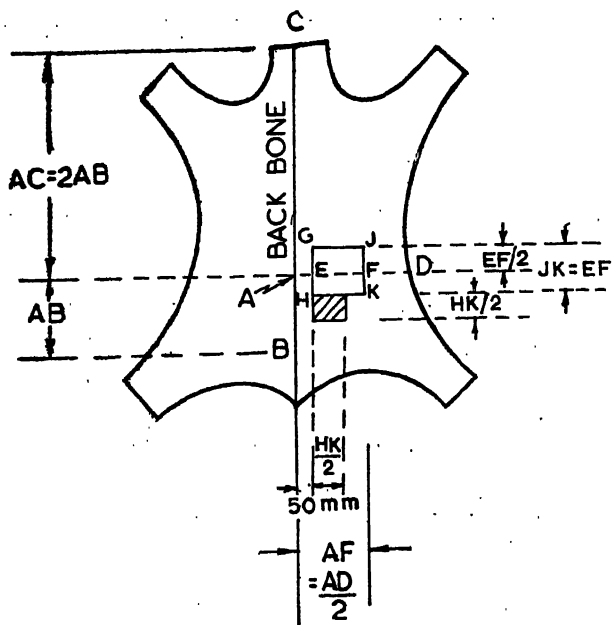
Sampling position of whole hides, sides and skins

FIG. 30
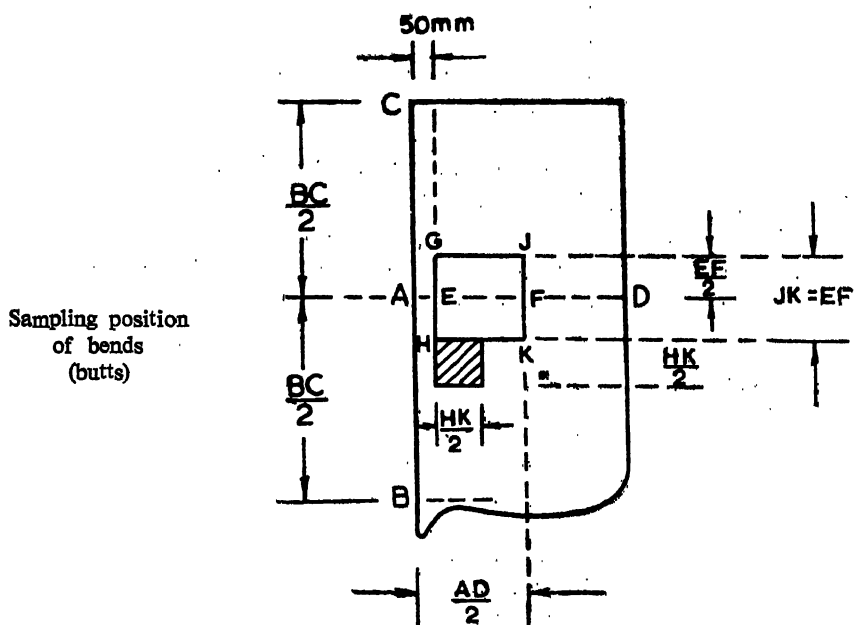
Sampling position of bends (butts)

FIG. 31

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For sampling bends (Figure 31), a square piece of leather is taken adjacent to the line HK, in the direction of the root of the tail, commencing from point H and with the side length equal to the half of HK. Figure 2 may be referred to for further details.

For sampling shoulders (Figure 32), a square piece of leather is taken adjacent to the line AB, in the direction of the head, commencing from the point A and with the side length equal to the half of AB. Figure 3 may be seen for additional particulars.

FIG.32.

Sampling position
of shoulders

FIG.33.

Sampling position of
bellies (flanks)

For sampling bellies, two square pieces of approximately 10 cm side length are taken along the cut edge TU, right and left from the central point Q of the side and immediately adjacent to the section envisaged for physical tests. Figure 4 provides more details.

A combined information on the physical and chemical characteristics of a particular sample of leather can be regarded as its bio-data which 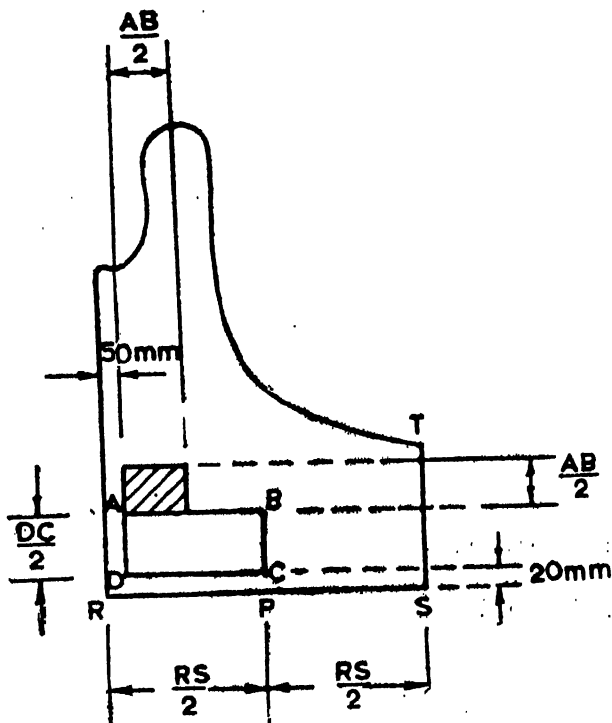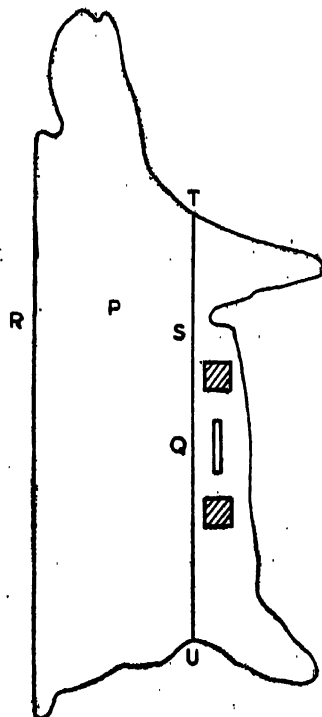forms the basis for its choice for the intended use. Leathers are formed by the combination of proteins with the tanning matter, constituting together the leather substance. Other components of leathers —moisture, extractable fatty matter, water soluble organic and inorganic matter, and water insoluble mineral matter—constitute the non-leather substance. Leathers tanned with specific mineral tanning agents contain that specific mineral matter.

The sample obtained through the sampling position of the leather is cut into pieces of convenient size to be fed into the wiley mill. The pieces of leather are then ground into powder in the wiley mill. In the succeeding paragraphs, 'the sample of leather for analysis' represents the leather powder thus obtained.

MOISTURE (LC 1/IS: 582—1970; SLC 3; IUC 5)

By moisture, it is generally meant the matter that is volatile at 102 ± 2 °C. This is best determined by heating the sampled powder in an air-oven maintained at 102 ± 2 °C for a minimum of 3 hours and till a constant weight of the dry matter is obtained.

$$\text{Moisture, per cent} = \frac{\text{Loss in weight of the leather (g)}}{\text{Weight of the leather taken for test (g)}} \times 100$$

OILS AND FATS (LC4/IS: 582—1970; SLC 4; IUC 4)

The term oils and fats signifies the matter extractable by dichloromethane (boiling point 38 —40 °C) or petroleum hydrocarbon solvent (boiling point 40 —60 °C). The matter thus extracted will contain the fatty substances present in the leather in the uncombined form, i.e., in the free condition.

The leather powder (10 g) is packed evenly in a suitable thimble (the thimlles made of No. 1 Whatman filter paper will serve the purpose); covered with a thin layer of grease-free non-absorbent cotton and placed in the middle extraction unit of the soxhlet apparatus. The free oils and fats are extracted thoroughly (usually for 3 hours with 5—6 minutes/cycle will be adequate for leathers containing normal amounts of greasy matter) using either dichloromethane or petroleum solvent. At the end of the extraction, the solvent is distilled off and the residue containing the extracted matter dried to constant weight at 102 ± 2°C over a period of 4—8 hours.*

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* The fatty substances extracted as above may be subjected to further analysis for various characteristics to obtain information on the nature of the oils contained in the leather.

LC—Methods of chemical testing of leathers.

SLC—Section: Chemical analysis of leather (all hides) published by the Society of leather Trades' chemists, UK.

IUC—Chemical analysis of leather published by the International Union of Leather Chemists' Society.

$$\text{Free oils and fats, per cent} = \frac{\text{Weight of residue (g)}}{10} \times 100$$

WATER SOLUBLES (LC6/IS: 582—1970; SLC 5; IUC 6)

The matter that is soluble in water at $27\pm2°C$ when the degreased leather powder is shaken up with (50 times the weight of original leather of) water in a mechanical shaker (50 ± 10 rpm) for 2 hours is termed as the water solubles. This represents both the soluble inorganic matter and the soluble organic matter, such as epsom salt, other mineral salts and glucose, unfixed vegetable tannins, etc., respectively.

The degreased leather powder remaining in the thimble after the soxhlet extraction for oils and fats is air-dried, transferred to a wide-mouthed glass bottle and 500 ml of distilled water are added to the bottle, closed with a rubber bung and the whole connected to the mechanical shaker and run for 2 hours. The bottle is then taken off from the shaker, the aqueous extract is filtered through a suitable filter paper (Whatman No. 11 or its equivalent), the first 50 ml of the filtrate rejected, 50 ml from the subsequent filtrate is pipetted into a tared evaporating basin, evaporated on a water bath, and the residue dried at $102\pm2°$ C in an air-oven to constant weight.*

Water solubles, per cent = Weight of residue (g) $\times$ 100

INSOLUBLE ASH (LC8/IS: 582—1970; SLC6; IUC 7)

The sulphated ash of the leather from which free oils and fats and water solubles are removed is called the insoluble ash or the water insoluble mineral matter. This represents inorganic substances (that are not soluble in water under the conditions of the test of water solubles) such as chrome, aluminium and other mineral matter fixed to the leather.

The leather after degreasing and after the removal of water soluble matter is air-dried, incinerated and ashed in a muffle furnace at 800°C and the resultant ash is cooled, moistened with 2N sulphuric acid, heated over a low flame and then at 800°C, cooled and weighed to constant weight.

Insoluble ash, per cent = Weight of ash (g) $\times$ 10

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* Aliquot of water solubles are used in the experiments for the desterminations of sulphated ash of water solubles , water soluble organic substances, pH of water solubles, difference, figure, water soluble magnesium-content, and glucose-content.

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The per cent insoluble ash is also expressed as the difference between the per cent total ash and the per cent sulphated ash of water solubles.

HIDE SUBSTANCE (LC5/IS: 582—1970; SLC7; IUC10)

The amount of proteinous matter, expressed in terms of collagen, the major protein of leather, is its hide substance.

About 1 g. of the prepared leather powder is weighed into a Kjeldahl flask (500 ml), a catalyst mixture containing about 10 g potassium sulphate and 0.1 g copper sulphate followed by 20 ml of concentrated sulphuric acid are added and the whole is digested in a fume cupboard. Loss of acid during the digestion may be curtailed by placing a small funnel in the neck of the flask. Upon completion of digestion, when the protein is converted into ammonium sulphate, the contents are cooled and diluted with about 150 ml water. 80 ml of 10N sodium hydroxide solution are then slowly added to the Kjeldahl flask down the sides, a few pieces of pumice stones are added, the flask is then connected to the distillation unit and the distilled-off ammonia vapours are absorbed into 100 ml of 4 per cent boric acid solution containing a suitable indicator that shows a colour change at pH 4.6 (mixed indicator containing 0.06 per cent methyl red and 0.04 per cent methylene blue in 96 per cent ethyl alcohol is found to be suitable.); the ammonia is then titrated against standard hydrochloric acid.

$$\text{Hide substance, per cent} = \frac{\text{Volume of acid (ml)} \times \text{Strength of acid (N)} \times 1.4 \times 5.62}{\text{Weight of leather (g)}}$$

FIXED ORGANIC MATTER (LC 21/IS: 582—1970; SLC12)

The per cent fixed organic matter is the difference between 100 and the sum of the percentages of moisture, free oils and fats, water solubles, insoluble ash and hide substance. This gives the amount of organic matter combined to the hide proteins—fixed tannins in the case of vegetable tanned leathers and fixed organic matter, i.e., organic anions used for masking mineral tanning agents, etc., in the case of such mineral tanned leather—, the organic matter existing in the free state having been accounted for earlier as the free oils and fats and part of the water solubles (organic matter soluble in water).

Fixed organic matter, per cent = 100 — (per cent moisture+per cent oils and fats+per cent water solubles + per cent insoluble ash + per cent hide substance)

or

100 — (per cent moisture + per cent oils and fats + per cent water soluble organic substances + per cent total ash + per cent hide substance)

LEATHER SUBSTANCE

The sum of the percentages of hide substance and fixed organic matter is called the per cent leather substance and is more appropriate to vegetable tanned leathers.

Leather substance, per cent = Per cent hide substance + Per cent fixed organic matter

DEGREE OF TANNAGE (LC21/IS: 582—1970; SLC12)

The amount of fixed tannins expressed on 100 parts of hide substance is the degree of tannage.

$$\text{Degree of tannage} = \frac{\text{Per cent fixed tannins}}{\text{Per cent hide substance}} \times 100$$

This characteristic being applicable only to straight vegetable tanned leathers, the term 'fixed tannins' is retained in the place of the general term 'fixed organic matter'. The degree of tannage is denoted only as a figure.

TOTAL ASH (LC 3/IS: 582—1970; SLC6; IUC7)

The amount of ash when the leather is ashed at 800°C and subsequently sulphated is termed the total ash.

About 5 g of the leather is first incinerated in a tared crucible, ashed in a muffle furnace at 800°C, the ash is cooled, moistened with 2N sulphuric acid, heated in a low flame and then at 800°C, cooled again and weighed to constant weight.

$$\text{Total ash, per cent} = \frac{\text{Weight of ash (g)}}{\text{Weight of leather (g)}} \times 100$$

This represents both the water soluble and water insoluble inorganic substances present in the leather.

SULPHATED ASH OF WATER SOLUBLES (LC7/IS:582—1970; SLC5; IUC6)

The ash of the water soluble inorganic substances, sulphated by treatment with sulphuric acid, is known as sulphated ash of water solubles.

An aliquot (50ml) of water solubles is evaporated on a water bath, the residue wetted with 2N sulphuric acid, heated over a low flame, and then ashed at 800°C, cooled and weighed to constant weight.

Sulphated ash of water
solubles, per cent   =   Weight of ash (g) × 100

This represents the water soluble inorganic substances contained in the leather.

WATER SOLUBLE ORGANIC SUBSTANCES (LC 9/IS: 582—1970; SLC5; IUC6)

The amount of water soluble organic substances is obtained as the difference between the water solubles and sulphated ash of water solubles.

Water soluble organic   =   Per cent water solubles—Per cent sulphated
substances, per cent   ash of water solubles

pH OF WATER SOLUBLES (LC18/IS:582—1970; SLC13; IUC11)

The pH of the aqueous extract containing the water soluble matter is found out using a pH meter with glass electrodes. This estimation is useful in assessing the acidity or alkalinity of the leather. See also footnote.

DIFFERENCE FIGURE (LC18/IS: 582—1970; SLC13; IUC 11)

The numerical difference between the pH values of water solubles and its ten-fold dilution is called the difference figure.

An aliquot of the water solubles, say, 25 ml is pipetted into a 250 ml standard flask and is then made up to the mark. The pH of the water solubles thus diluted

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If the pH of water solubles and the difference figure are only to be determined and not the amount of water solubles as such, the acqueous extract of the leather could be obtained by shaking 5 g of leather powder with 100 ml water for 16-24 hours and the unfiltered extract then taken for the determination of pH and for dilution towards the estimation of difference figure; in the case of highly greasy leathers, it is advisable to remove the fatty matter in the soxhlet apparatus, air-dry the leather, and then take up the leather for the determination of pH.

is determined with a pH meter with glass electrodes. The difference between the pH of original water solubles and that of the diluted water solubles gives the difference figure.

For leathers whose pH values of water solubles are less than 4 and more than 10, the difference figure will be higher if the leather contained strong acids or strong bases and in the absence of buffering salts; the difference figure will be smaller when the strong acids or strong bases are present along with considerable quantities of buffering salts or in the presence of weak acids or weak bases independent of buffering salts. Usually, the estimation of difference figure is appropriate only when the pH of water solubles is below 4 and above 10. The pH value of water solubles and the difference figure serve to measure the acidity or alkalinity of the leather and are applicable for all types of leathers in all stages of processing.

CHROME-CONTENT (LC10/IS; 582—1970; SLC8; IUC8)

The chromium in the leathers is estimated either through wet oxidation method or by fusion with oxidising mixtures and expressed as chromic oxide (Cr_2O_3).

In the wet oxidation method, the leather powder (1 g) is digested with an acid-mixture (5 ml conc. nitric acid, 5 ml conc. sulphuric acid and 10 ml of 60 per cent weight by volume perchloric acid) when the green-coloured trivalent chrome gets oxidised to orange-coloured hexavalent state. Upon ensuring that the oxidation is complete (after the change in colour, gentle and controlled heating for two minutes is usually sufficient), the whole mixture is rapidly cooled to avoid loss of chromic acid, diluted to about 150 ml, the excess free chlorine is boiled off during a period of 10 minutes (a few pieces of pumice stones may be added to prevent bumping), cooled and titrated with standard sodium thiosulphate solution in the presence of 20 ml of 10 per cent potassium iodide solution and using starch solution as the indicator towards the end. Iron, if any, may be masked with an addition of 15 ml of 85 per cent (weight by volume) phosphoric acid prior to the addition of potassium iodide while starting the titration.

Chrome content, as Cr_2O_3, per cent = Volume of thiosulphate (ml) × Strength of thiosulphate (N) × 2.534

In the fusion method, the oxidation of chrome is accomplished with either 1:1:1 anhydrous sodium carbonate: potassium carbonate: sodium tetraborate or 1:1 anhydrous sodium carbonate: potassium chlorate. The leather is first ashed, fused with either of the oxidising mixtures, the melt is acidified and the hexavalent chrome is titrated with standard sodium thiosulphate as usual.

ALUMINIUM-CONTENT (LC11/IS: 582—1970)

The aluminium in the leathers can be estimated volumetrically and is expressed as Al_2O_3. The sample of leather (1 g) is ashed at 800°C in a muffle furnace, lixi-

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viated with 5 ml conc. hydrochloric acid followed by distilled water, heated over a gentle flame to reduce the volume to about 5 ml., a few drops of mixed indicator (0.1 per cent solutions of thymol blue and cresol red in the ratio of 3:1 in alcohol) are added, the excess acid is neutralised with 5N potassium hydroxide solution (disappearance of red colour), the solution cooled, 20 ml of 50 per cent solution of potassium tartrate added and the solution brought to exact neutral point (appearance of pink colour) by using 0.1N potassium hydroxide solution. At this stage, 20 ml of 20 per cent potassium fluoride solution (previously made neutral to the mixed indicator: pink colour) are added which liberate an amount of potassium hydroxide that is equivalent to the aluminium from the aluminium-tartrate complex. The liberated potassium hydroxide is then titrated with standard hydrochloric acid to neutral point (apperance of pink colour).

$$\text{Aluminium content, as } Al_2O_3, \text{ per cent} = \text{Volume of hydrochloric acid (ml)} \times \text{Strength of hydrochloric acid (N)} \times 1.7$$

TOTAL CHLORIDES (LC19/IS: 582—1970)

The amount of chlorides present in the leather is estimated and expressed as sodium chloride.

The leather sample (10 g) is first incinerated and then ashed in a muffle furnace at 550°C. The ash is moistened with conc. nitric acid and made up to 500 ml. An aliquot, say 100 ml, is then taken for the chloride estimation through argentimetry by neutralising the acid with the addition of solid calcium carbonate and titrating with standard silver nitrate solution using a 5 per cent solution of potassium chromate as the indicator.

$$\text{Total chlorides, as } NaCl, \text{ per cent} = \text{Volume of } AgNO_3 \text{ (ml)} \times \text{Strength of } AgNO_3 \text{ (N)} \times 2.923$$

TOTAL SULPHATES (LC20/IS: 582—1970)

The amount of sulphates present in the leather is estimated and expressed as sodium sulphate.

Another aliquot from the determination of total chlorides, say 100 ml, is acidified with 10 ml of conc. hydrochloric acid, quantitatively filtered using about 250 ml water, heated to boiling, and the sulphates precipitated with 10 ml of hot 12 per cent barium chloride solution. The precipitated solution is kept on a water bath for half-an-hour and allowed to stand overnight. The precipitate of barium

sulphate is quantitatively filtered using either a gooch crucible or Whatman No. 42 gravimetric filter paper, washed free of chlorides, dried and ignited to constant weight.

Total sulphates, as $Na_2 SO_4$, per cent = Weight of residue (g) $\times$ 30.47

In addition to the preceding determinations, sometimes certain special characteristics may have to be estimated in the leathers and the broad working principles for such estimations are provided in the following.

COMBINED OILS (LC15/IS: 582—1970)

A portion of the oils and fatty matter is present in the leather in combined form which could not be extracted directly as in oils and fats described earlier. This combined fatty matter is determined through alkali-digestion of the leather, when the fatty matter is converted into soaps which are then transformed into fatty acids by the addition of mineral acid and the fatty acids thus formed are then taken up with solvents. The free oils and fats present in 10 g. of the leather sample are extracted by either dichloromethane or petroleum hydrocarbon solvent thoroughly. The degreased leather is then digested with 50 ml of 1.5N alcoholic potassium hydroxide in a refluxing unit, alcohol evaporated off, the residue diluted to 50 ml with distilled water, the resulting soap solution is then acidified with 15 ml of conc. hydrochloric acid and heated on a water bath for 1 hour when the soaps get converted into fatty acids. The fatty acids are then successively extracted with diethyl ether in a separating funnel. The combined ether extracts are washed with water, free of mineral acids, dried over anhydrous sodium sulphate, filtered into a tared flask, ether evaporated off, and the residue dried at 100 $\pm$ 2°C to constant weight in an air-oven.

Combined oil, per cent = Weight of residue (g) $\times$ 10

EPSOM SALT (LC16/IS: 582—1970; SLC15; IUC9)

The epsom salt content of the leather is determined and expressed as $MgSO_4, 7H_2O$. While the total magnesium content is determined on the total ash of the leather, the water soluble magnesium content is determined on sulphated ash of water solubles. The interfering phosphate and calcium ions, if any, should be successively eliminated using ferric chloride and ammonium oxalate respectively. Magnesium can then be estimated by complexometric method using the solution of disodium salt of ethylene-diamine tetra acetic acid, Eriochrome Black-T indicator and ammonia-ammonium chloride buffer solution, the end point of the titration being a distinct change of colour to pure blue. Each ml of 0.01M solution of disodium salt of

. EDTA corresponds to 0.002465 g. of MgSO₄, 7H₂O, which is then expressed as a percentage by weight of the sample of leather. The estimation of epsom salt content gives an idea of the extent of adulteration, if any, of the leather.

GLUCOSE (LC17/IS; 582—1970; SLC14 ·

The glucose content of the leather is determined from its aqueous extract. An aliquot of the aqueous extract is treated with a saturated solution of lead acetate to remove the tannins, excess lead is precipitated as lead oxalate using solid potassium oxalate, the sugars in the tannins-free solution thus obtained are converted into reducing sugars by refluxing with hydrochloric acid, the solution cooled, the excess acid neutralised with sodium carbonate, and made up to a convenient volume. An aliquot of the made up solution is boiled with Fehling's solution and the resultant cuprous oxide filtered, washed successively with hot water, rectified spirit and ether, dried and weighed. Under prescribed conditions of the experiment with respect to dilutions, duration of boiling with Fehling's solution, drying of Cu₂O, etc., the values of glucose can be read off for various figures of Cu₂O from the Munson and Walker's table. The glucose content is then expressed as a percentage on the weight of the leather sample. This experiment also indicates whether the leather is adulterated with excessive amounts of glucose.

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APPENDIX-I

RECOMMENDED QUALITY REQUIREMENTS FOR THE MAIN TYPES OF LEATHER

Requirement	Shoe upper leather								Sole leather				Lining leather		
	Box and willow calf	Box and willow side	Corrected grain side	Glazed kid	Waterproof (combination-tanned)	Waterproof (chrome tanned)	Vegetable-tanned upper leather	Suede (cattle, calf, goat, split)	Sole leather modern tannage	Sole leather old pit tannage	Insole leather	Insole, sock lining (sheep), combination tanned	Vegetable tanned	Combination tanned	Chrome tanned
Max. ash (%) (after subtracting tanning oxide)	2.0	2.0	2.0	2.0	2.0	2.0	1.0	2.0	3.0 (epsom salt 4.0)	0	2.5 (epsom salt 4.0)	2.0	2.0	2.0	2.0
Min. chromium compounds (Cr_2O_3) (%)	2.5	2.5	2.5	2.5	0.5	2.5		2.5				0.8			2.5
Fatty substances (%)	2.6	min. 2.0	min. 2.0	4,8	17-23	8-15	16-23	2-6	max. 3.0	max. 0.7	max. 4.0	max. 4.0	3-12	0.5	2.5
Max. loss by washing (%)					6.0	6.0		6.0	16.0	6.0	10.0	10.0	6.0	3.0	
Degree of tannage					min. 30	min. 50	min. 50	min. 50	60-95	60-95	60-95	min. 50	min. 50	min. 40	
pH	aqueous extract (1:20) not below 3.5													3-12	3-12
Difference value	at pH values below 4.0 difference value not above 0.70														
Min. tensile strength (kg/cm²)	200	200	200	200	250	300	250	200	200	200	200	100	150	150	150

Property															
Max. elongation at break (%)	80	80	80	80	80	80	80	75	30	35	35	40	70	100	150
Min. stitch-tear strength (kg/cm.)	80	100	80	60	100	120	100	80	100	130	100		40	40	40
Min. split-tear strength (kg/cm.)	30	40	25	25	40	40	40								40
Water absorption (%) after 2 hours	max. 60	max. 60	max. 60	max. 60	max. 60	max. 30	max. 30	max. 35	max. 40	40	25	min. 100	min. 100	100	100
after 24 hours	max. 85	max. 85	max. 85	max. 85	max. 85	max. 45	max. 45	max. 45	max. 50	50					
Max. volume weight (g/cm³)	1.15								1.15	1.05	1.00		1.00		
Min. air permeability value (cm/min per cm Hg)	80	80	80	80	80	80	80	80	20	20	20	250	250	250	
Min. water-vapour permeability value (mg/cm²)	250	250	250	250	180	200	200	250	200	200	250		300	300	300
Max. wear coefficient (%)	3.0								2.0						

Requirement	Harness, bag and upholstery leather (vegetable tanned)	Harness, bag and upholstery leather (combination tanned)	Harness, bag and upholstery leather (chrome tanned)	Fancy leather (vegetable tanned)	Clothing leather (chrome tanned)	Glove leather (chrome tanned)	Glove leather (alum tanned)	Hot sweat band leather (vegetable tanned)	Oil-tanned leather	Harness and belt leather (vegetable tanned)	Harness and belt leather (chrome tanned)	Football leather (chrome tanned)	Leather for workers protective articles (chrome tanned)	Raw hide and transparent leather	Chamois leather
	(Upholstery and fancy leather)				Clothing leather				Technical leather						
Max. ash (%) (after subtracting tanning oxides)	2.0	2.0	2.0	2.0	2.0	2.0	8.0 Al_2O_3	2.0 Al_2O_3	6.0 Al_2O_3	2.0	2.0	2.0	2.0	1.5	5.0
Min. chromium compounds (Cr_2O_3) (%)		0.8	2.5		2.5	2.5	min. 2.0		min. 1.0		2.5	2.5	3.0		
Fatty substances (%)	3-12	3-12	3-12	3-8	4-10	4-10	max. 10	3-8	max. 35	10-25	10-25	4-10	4-15		max. 10 (fixed 0.5-3.0)
Max. loss by washing (%)	6.0	6.0		6.0				4.0		6.0				4.0 max.	
Min. degree of tannage	50	30		50				50		50					
pH	aqueous extract (1:20) not below 3.5													8.5	8.0
Difference value	at pH values below 4.0 difference value not above 0.70														
Min. tensile strength (kg/cm²)	above 2 mm 250 below 2 mm 100	above 2 mm 250 below 2 mm 100	above 2 mm 250 below 2 mm 150	100	100	100	100	100	350	250	250	300	200	600	100

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	max.	max.	max.	max.	max.	min.	max.	max.	max.	max.	max.	max.	max.	max.	min.
Elongation at break (%)	50	50	100	50	60	50	50	90	50	50	75	70	75	35–60	50
Min. stitch-tear strength (kg/cm)	above 2 mm 100 / below 2 mm 30	above 2 mm 50 / below 2 mm 30		25	60	40					100	120			40
Min. split-tear strength (kg/cm)	above 2 mm 40 / below 2 mm 15	above 2 mm 20 / below 2 mm 15	10	15	25	25	10		40	40	40		15		15
Water uptake (%)						after 5' min. 100 / after 2h min. 130				after 2h max. 35 / after 24h max. 70					
Water-vapour permeability value (mg/cm²)	min. 250														

SOURCE:— *Aceptable Quality Levels in Leathers: Maintaining Quality Control in Semi-processed and Finished Leathers Produced in Developing Countries for Export*, (1976) *United* Nations Industrial Development Organization (Vienna), United Nations, New York, USA.

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APPENDIX 2

SELECT LIST OF SUPPLIERS OF PHYSICAL TESTING INSTRUMENTS

Physical/characteristic	*Address of the supplier*
TENSILE TESTER	
Tensile strength	(i) Scott Testers Inc., Providence, R.I· USA.
Tearing strength	
Tongue tear strength	(ii) Precision Scientific Equipment Corporation
Stitch tear strength (double hole)	16, Patel Road, South Patel Nagar, New Delhi - 110 008
Buckle tear strength	India.
Split tear strength	(iii) Kamal Metal Industries, Gajjar House
Elongation at break	Astodia Road
Elongation at a specified load	Ahmedabad I.
	India.
GRAIN CRACK TESTER	
Grain crack index	H.W. Wallace & Co. Ltd.
Extension at grain crack	126, Wellesley Road
	Croydon
	Surrey
	England
	(1) MULLEN'S TESTER AND (2) LASTOMETER
Grain crack strength	(1) B.F. Perkins & Sons Inc.
	Holyoke
	Massachussetts
	USA.

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Barsting strength

(2) (i) Baird & Taltock (London) Ltd.
14/17 St. Cross Street
London, England

(ii) Shoe & Allied Trades Research Association
SATRA house
Rockingham Road
Kettering
Northants NN 16 9 JH
England

TENSOMETER

Two-dimensional extension: (1) area extension; (2) radial extension and (3) linear tension

Bally Schuhfabriken AG
CH 5012
Schonenwerd
Switzerland

RUBBER ABRADER CAT. NO. 9D 400

Resistance to abrasion of heavy leather

American Instrument Co. Inc.
Silver Spring
Maryland
Washington D.C.
USA

TABER ABRASER

Resistance to abrasion of light leather

Taber Instrument Corporation
North Tonawanda
New York
USA

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KUBELKA APPARATUS

Water absorption (static)

Central Leather Research Institute
Adyar
Madras 600 020
India

PERMEOMETER

Waterproofness of sole leather (dynamic): (1) penetration time;
(2) water absorption and (3) penetration rate

Bally Schuhfabriken AG
CH 5012
Schonenwerd
Switzerland

PENETROMETER

Water proofness of upper leather (dynamic): (1) penetration time;
(2) water absorption and (3) transmission rate

Bally Schuhfabriken AG
CH 5012
Schonenwerd
Switzerland

WATER VAPOUR PERMEABILITY APPARATUS

Water vapour permeability

H.W. Wallace & Co. Ltd.
St. Jame's Road
Croydon
Surrey
England

C & R TESTER

Compressibility and resilience

Custom Scientific Instruments Inc.
541 Devon Street
Kearny
New Jersey
USA

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DRAPE METER

Drape coefficient — Bombay Textile Research Association
Lal Bahadur Shastri Marg
Ghatkopar (West)
Bombay—400 086   India

SHRINKAGE TESTER

Shrinkage temperature — (i) Arthur H. Thomas Co.
Philadelphia Pa
USA

(ii) Scientific Engineering Co.
19, Ramachandra Reddiar Street
Ponneri 601 204
Chingleput District, Tamil Nadu, India

(1) FLEXOMETER (BALLY) AND (2) FLEXOMETER (GEORGES PERRIN)

Resistance to flexing —

(1) Bally Schuhfabriken AG
CH 5012
Schonenwerd
Switzerland

(2) Georges Perrin
22 rue Branly
69 Villeurbanne (Rhone)
Lyon, France

FINISH RUB FASTNESS TESTER

Fastness to rubbing — Shoe & Allied Trades Research Association
SATRA house
Rockingham Road
Kettering
Northants NN 16 9 JH
England

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GREY SCALE FOR ASSESSING CHANGE IN COLOUR AND GREY SCALE FOR ASSESSING STAINING

Fastness to perspiration

(i) British Standards Institution
British Standards House
2 Park Street
London W1Y 4AA
England

(ii) Indian Standards Institution
Manak Bhavan
9 Bahadur Shah Zafar Marg
New Delhi-110 002
India

(1) WOOL BLUE STANDARDS, GREY SCALE FOR ASSESSING CHANGE IN COLOUR AND GREY SCALE FOR ASSESSING STAINING AND (2) XENOTEST

Fastness to light

Fastness to weather

(1) (i) British Standards Institution
British Standards House
2 Park Street
London W1Y 4AA
England

(ii) Indian Standards Institution
Manak Bhavan
9 Bahadur Shah Zafar Marg
New Delhi 110 002
India

(2) Original Hanau
Quarzlampen GmbH
6450 Hanau
Hohensonne strasse
W. Germany

THERMOTEST

Resistance to heat of finish layer

Setaram
101-103 rue de Seze
69 Lyon 6e
France

ENVIRONMENTAL TEST CHAMBER (TYPE DF 2/70)

Resistance to cold temperature

South London Electrical Equipment Co. Ltd.
Lanier Works
Hithergreen Lane
London SE13 6QD
England